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BY
ALFRED GOLDBERG

American Iron & Steel Institute
150 East Forty-Second Street
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Attention: Mr. Charles M. Parker, Assistant Vice President

Dear Sir:

Attached hereto is a technical report which has been prepared for distribution. The results described herein pertain to some of the studies made on the investigation "The Interrelation Between Creep and Microstructural Instabilities".

The cooperation and support given by the American Iron and Steel Institute in making these studies possible are sincerely appreciated. The author also would like to acknowlege the interest shown by the U. S. Naval Postgraduate School and the Office of Naval Research in supporting the development of the laboratory facilities for this general area of research.

Respectfully submitted

THE SOFTENING BEHAVIOR OF A COLD-ROLLED LOW CARBON STEEL UNDER STATIC AND DYNAMIC ANNEALING CONDITIONS

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ABSTRACT

The results of an investigation on the softening behavior of a cold-rolled low carbon steel are presented. The analyses are based on hardness and microscopic observations. Specimens, originally cold rolled 15, 30, 50, and 75% reduction in thickness, were annealed at several temperatures ranging from 481 to 687°C. The effect of a concurrent load on the softening behavior was also investigated by applying a tensile creep load, during annealing, on a number of 50% cold-rolled specimens.

Three important competing processes appear to control the annealing kinetics when no load is applied, namely, recovery, polygonization, and recrystallization. A fourth process, that of vacancy declustering, appears to play a definite, although minor, role in the annealing of a cold-worked metal subjected to a small amount of concurrent plastic straining. With larger strains polygonization is introduced or accelerated. Under such conditions the recovery or recrystallization process which would take place under static annealing is interrupted. Other than this interruption, however, the latter two processes are not affected by concurrent straining. A qualitative consideration of the effect of material and test variables on determining which of the competing processes dominates during annealing, is presented.

INTRODUCTION

The softening behavior of a cold-worked metal, classically depicted as occurring by recovery, recrystallization, and grain growth, has been the subject of numerous investigations. The literature dealing with these phenomena has been reviewed in several survey papers(1-5)*. During the past decade a new softening mechanism, that of the formation of low-angle boundary substructures, referred to as polygonization, has been receiving continually more attention(6-9). Under certain conditions complete elimination of the strain energy of cold work may take place by this process(7,9-11). The development of such substructures and their contribution to strain, during elevated-temperature deformation of both cold-worked and annealed metals, have been reported on frequently(12-15). The appearance of fine substructures resulting directly from cold working also has been observed(4,16). A concept, which has received some attention, that of the presence of relatively strain-free 'preformed' regions subsequently acting as nuclei in the annealing of a cold-worked metal, is supported by the existence of such cold-worked substructures(4). These strain-free regions are depicted as being bounded by zones of severe lattice curvature containing a large excess of dislocations of the same sign(16).

Recent discussions on softening, especially pertaining to recovery and polygonization, and to a minor extent to recrystallization, are concerned largely with dislocation movement resulting in modifications in the pattern and density of dislocations which existed in the cold-worked state(4). More recently, studies have been made on a new type of crystal defect, lattice vacancies, which appears to play a secondary, though important, role in the strengthening properties of

*The figures appearing in parentheses pertain to the references appended to this paper.

metals[17]. The coalescence of vacancies, i.e., vacancy clustering[18], their partial elimination, and their interaction with other lattice defects, such as with dislocations, all of which may take place during annealing, especially in the recovery range, must also be taken into account when analyzing the softening behavior of a cold-worked metal.

This paper deals with experimental observations of the softening behavior of a cold-worked low carbon steel, especially when under the influence of an externally applied stress. The results obtained can be explained on the basis of the competing roles of the above-mentioned phenomena which may take place during annealing of a cold-worked metal. Throughout this paper the term 'recrystallization' is used in the classical sense, i.e., the formation and growth of strain-free nuclei. Here, the originally deformed grains lose their identity; the interface consists of high-angle boundaries. 'Polygonization' refers to the change in hardness associated with low-angle subboundary formation and movement. When neither recrystallization or polygonization are observed, as viewed by ordinary microscopic techniques, the annealing phenomena will be referred to as 'recovery'. When a load is applied on the specimen resulting in concurrent plastic straining during annealing the term 'dynamic annealing' is used. The term 'static annealing' is used for conventional annealing, i.e., softening without the application of any load.

TEST MATERIAL AND EXPERIMENTAL PROCEDURE

An aluminum-killed low carbon steel was used as the test material. The steel was kindly supplied by Kaiser Steel Corporation; its chemical composition is given in Table I. This material was received in the form of hot-rolled 1/8-inch thick sheets which were cut in the rolling direction into 3/4-inch wide strips, then pickled and cleaned in preparation for cold rolling. The strips were cold rolled 15, 30, 50 and 75 percent reduction in thickness. The hardness values of the four

cold-rolled states as well as the initial hot-rolled hardness are shown in Table II. Due to the lack in thickness of the more severely reduced strips, the Rockwell Superficial Hardness Scale was used in all cases. The cold-rolled strips were cut transversely into 3/16-inch by 3/4-inch coupons for the static annealing studies. Data on dynamic annealing were obtained from tensile-creep specimens having a 1/4-inch wide reduced section machined from 4½-inch long blanks cut from the same strips. Where only small strains were desired, the blanks, with 1/4-inch gripping-pin holes at each end, were used directly.

TABLE I

CHECK ANALYSIS MADE BY KAISER STEEL CORPORATION

Element	C	Mn	P	S	Si	Cu	Ni	Cr	V	Mo	Co	Al
Percent	.06	.34	.010	.030	.01	.08	.02	.01	.005	.005	.035	.012

To avoid oxidation, and to minimize and control the time required to reach the annealing temperature, a stirred-liquid bath consisting of a ternary carbonate eutectic was used as the heating medium. Temperature control of better than

TABLE II

HARDNESS VALUES OF SPECIMENS IN DIFFERENT INITIAL CONDITIONS

CONDITION	ROCKWELL 45T SUPERFICIAL SCALE
As hot-rolled	43±3
15 percent cold-rolled	61±1
30 percent cold-rolled	68±1
50 percent cold-rolled	71.5±1
75 percent cold-rolled	74±1

±1°C, of the value reported in each case, was maintained during the test period with a variation of less than 1/2°C throughout the bath. After immersion in the salt, bath temperatures were reached within 30 seconds and 2 minutes for the coupons and mounted tensile specimens, respectively. This was ascertained during some trial runs by inserting a fine insulated thermocouple wire into a hole drilled parallel to the specimen surface.

Specimens were air cooled from the test temperature. The salt, which had solidified around a specimen, was dissolved by dipping in a 5% acetic acid solution. The effect of annealing was followed first by hardness measurements. Extensive microscopic observations were subsequently made to supplement and explain the hardness results. Specimens were etched by several alternate immersions in 65% saturated picric acid in ethyl alcohol and 1% nital.

PRESENTATION AND DISCUSSION OF EXPERIMENTAL RESULTS

A. Static Annealing Tests

The softening kinetics, as depicted by a loss of hardness for specimens, in three different initial cold-worked states annealed at various temperatures, are shown in Figs. 1-3. Three different softening behaviors are apparent. One, which is especially obvious at high temperatures and more prevalent for the severely cold-worked state, reveals a sharp inverse S-shaped curve typical of a nucleating-type process and what might be expected if softening occurs predominantly by recrystallization. All the curves in Fig. 1, the four curves furthest to the left in Fig. 2 and the highest temperature curves in Fig. 3 are typical of this type of behavior. Extensive microscopic examinations revealed that these curves can be accounted for completely on the basis of a recrystallization process. The region prior to the rapid drop in hardness would also be associated with recovery as well as the incubation period for recrystallization. Figs. 4 and 5 show photomicrographs depicting typical stages in recrystallization for the 75% and 50% cold-rolled states at 597°C. The change in microstructure closely parallels the drop in hardness shown in Figs. 1 and 2.

A second type of softening behavior is depicted by the curves obtained for specimens annealed at the lower temperatures, expecially for the lesser cold-worked states, as illustrated by the 481°C curve in Fig. 2 and the 481, 510 and 542°C curves in Fig. 3. The kinetics, here, are typical of a non-nucleating-type

process. Microstructure examinations of specimens annealed in this region suggest that certain changes from the cold-worked state take place almost instantaneously on reaching temperature; additional time at temperature apparently has virtually no effect on further modifying this initially-annealed microstructure. An example of this may be seen in Fig. 6, which contains the 50% cold-rolled structure together with those obtained after annealing for 0.10 hours and 818 hours at 481 °C, at a magnification of 2000. The start of a rapid drop in hardness at about 2500 hours indicating the initiation of recrystallization is suggested here in Fig. 2. Reference to this will be made in a later section. Fig. 6A is typical of a deformed structure consisting of distorted elongated grains containing numerous deformation bands and areas of cloudiness; the latter is probably associated with regions of severe lattice bending containing a high density of dislocations in rather diffuse arrays. This cloudiness was observed to be eliminated almost instantaneously, and certainly within one minute, on reaching the annealing temperature, suggesting a sudden release of piled-up dislocations away from the severely bent regions. The absence of any significant change in microstructure with additional annealing time may be seen by comparing Figs. 6B and C. Both microstructures consist of elongated grains containing deformation bands. An occasional lineal structure may be seen suggesting that perhaps substructures are forming and thus polygonization is in progress. However, the nature and extent of these markings are the same following annealing periods of 0.1 hours and 818 hours, although a hardness drop is obtained during this extensive time interval. Thus, aside from the initial change, it would appear, when using conventional microscopic techniques, that the phenomena largely responsible for softening in this region do not give rise to any microscopic changes and, accordingly, will be referred to as recovery.

Although the regions in which either recrystallization or recovery predominates appear reasonably well defined, an intermediate region exists in which

an erratic softening behavior is observed. In Fig. 2 the data at 510°C for the 50% cold-rolled condition show considerable scatter, while the curve at 523°C appears to be levelling off at a higher value of hardness than was obtained at the higher temperatures. A similar pattern may be seen in Fig. 3 at 597 and 610°C for the 15% cold-rolled condition. The general shape of the curves which contain considerable scatter suggests the presence here of a non-nucleating-type of process. Extensive microscopic examination, especially of the 50% cold-rolled specimens annealed at 510°C, showed that the annealing behavior here is modified due to the presence of polygonization. Typical microstructures obtained after annealing in this region together with those obtained at higher temperatures are shown in Figs. 7 and 8 at magnifications of 2000 and 750, respectively. The specimens represented in these photomicrographs have approximately the same hardness corresponding to a drop of about 55% from the cold-rolled value.

Detailed differences resulting from a 597°C and 510°C anneal are evident in Fig. 7. The specimen annealed at 597°C (Fig. 7A) shows the presence of recrystallized and elongated grains typical of what may be expected from interrupting a recrystallization process. The still-deformed elongated grains reveal a fine network of deformation bands which have replaced the cloudy structure observed in the as cold-rolled state (Fig. 6A). This latter type of change is similar to what was observed for the recovery region (Figs. 6B and C). The softening at this temperature is predominantly due to the formation of new strain-free grains. Annealing at 510°C, to essentially the same drop in hardness, results in the formation of a substructure which has developed to various degrees within different grains (Figs. 7B and C). The more well-defined and larger substructures result in lighter etching areas. In some instances the substructure may be presumed to occupy a complete grain. In general, the elongated pattern resulting from the previous deformation is retained. The latter is seen more clearly at a lower magnification shown in Fig. 8F. A general rumpling of the elongated grain

[illegible]

boundaries may be seen, which probably results from surface tension effects of the individual subgrains bordering the boundaries. The softening at 510°C, thus, would appear to take place primarily by the process of polygonization, whereby the strain energy of cold-rolling is eliminated by the formation and growth of substructures within the originally deformed grains.

A more general observation of the change in microstructure in going from the recrystallization to the polygonization region may be obtained from Fig. 8. The microstructures for the recrystallization region, 597 to 542°C (A-D) are very similar containing somewhat over 50% recrystallized grains. At 510°C (F), however, about 95% of the grains are still elongated, although the hardness is the same. Various degrees of clearness which, at a magnification of 2000, were associated with the development of the substructure, may be observed here. The few grains present which are not elongated are probably the same as those seen at the higher magnification which are similarly shaped and void of any substructure. It would be difficult to ascertain whether these grains resulted directly from recrystallization or from polygonization. Nevertheless, from the large proportion of elongated grains seen at the lower magnification together with the substructure shown at the higher magnification it is evident, as suggested in the above, that softening at 510°C occurs primarily by polygonization. At 523°C the microstructure (Fig. 8E) is somewhat in between those observed where recrystallization and polygonization are respectively predominant. This is consistent with the corresponding annealing curves shown in Fig. 2 in which the softening kinetics may be seen to be similar over the annealing range of 597 to 542°C. At 523°C the data appear to level off more gradually and at a higher value; some scatter is evident. At 510°C the kinetics become erratic and result in considerable scatter.

B. Dynamic Annealing Tests

The effect of dynamic annealing on modifying the softening kinetics observed during static annealing was studied in some detail for the 50% cold-rolled con-

dition at three temperatures, 542, 510, and 481°C where recrystallization, polygonization, and recovery predominate, respectively. The change in hardness with time for the dynamic and static annealing specimens tested at 542°C are compared in Fig. 9. The dynamic annealing tests were made by applying a constant load to tensile-type creep specimens giving initial creep stresses ranging from 7,500 to 27,500 psi. The tests were interrupted for room-temperature hardness measurements.

The effect of dynamic annealing is first to displace the static annealing curve to lower hardness values. The data for the three lowest stresses (7,500-15,200 psi), however, may be seen to fall within a common band. Furthermore, the values within this band run essentially parallel to the static annealing curve, even to the extent that the time for the onset of recrystallization, at approximately 0.45 hours, is the same in both cases. It appears that the phenomenon responsible for the extra softening arising from dynamic annealing, under the lower stresses, does not interfere with recrystallization. With initial increased amounts of concurrent straining, as would be obtained under the higher stresses of 20,000 and 27,500 psi, the hardness is displaced to still lower values. In addition, at these higher stresses and with increased straining over longer time intervals at the lower stresses the shape of the annealing curve changes with respect to the static annealing curve. This suggests the presence of other phenomena which may play an important role in interrupting or altering the recrystallization kinetics. These changes, which may be attributed to creep per se, will be discussed later. Of more immediate interest are the regions associated with small concurrent strains, avoiding the complications of creep.

Microstructures of static and dynamic-annealed specimens are shown in Fig. 10. A comparison of the static-annealed specimens (A-B) with those obtained by dynamic annealing under a low stress (D-E) reveals no significant difference in microstructure. The early additional drop in hardness, arising from the presence

of small concurrent straining during dynamic annealing shown in Fig. 9, is therefore to be associated with some submicroscopic change. To attempt an explanation of any such phenomenon one must analyze the type of hardening defects created during cold-working. Furthermore, these defects must be susceptible to rapid modifications by the moving dislocations which accomodate concurrent straining during the early dynamic annealing period.

It is currently believed that many lattice vacancies are created during plastic deformation at low temperatures, possibly from dislocation intersection processes of the screw-screw type [19]. These vacancies may be present as isolated vacancies or they may agglomerate by diffusion and form vacancy clusters [18,20]. An additional method of introducing vacancies is to quench from a temperature near the melting point, the equilibrium vacancy concentration increasing with temperature [21]. Experimental evidence indicating the presence of aggregations or clusters of vacancies have been convincingly presented using transmission electron microscopy [18]. The strength of a metal is believed to be increased by the presence of these clusters [20,22] which may be considered to act as obstacles to dislocation motion in much the same way as precipitates. Experimental evidence suggesting hardening due to the presence of quenched-in vacancies has been presented by Kimura, et al [23].

It is possible that the sudden relative drop in hardness under dynamic annealing is due to the elimination of vacancy clusters by moving dislocations as they impinge on these clusters. The clusters, which at lower temperatures normally act as obstacles, may disappear by acting as sources of vacancies for the impinging dislocations; the vacancies would permit the dislocations to climb away from these obstacles, the latter, in turn, being eliminated. This behavior is consistent with reported results which showed that small plastic deformations imposed on quenched single crystals of aluminum greatly accelerated the rate of recovery of electrical resistivity during annealing [24]. The high resistivity

in the as-quenched crystal was ascribed to the excess amount of vacancies retained upon cooling.

If clusters act in the same way as precipitates, then the effect of their elimination should be manifested in reduced strain hardening resulting from the removal of such obstacles to the movement of dislocations during plastic deformation. The results of our hardness tests, which in a sense are a measure of resistance to plastic deformation, are in agreement with this concept. However, the impinging dislocations may interact with some of the residual vacancies to form a stable atmosphere of vacancies in much the same way as was orginally proposed by Cottrell for solute-atom locking of dislocations [25]. If this were to happen, the metal should exhibit a more marked yield point even though its resistance to subsequent plastic deformation be reduced as a result of the dynamic annealing. The tests described below were performed to examine this possibility.

Four tensile specimens were cut from a 50% cold-rolled strip. One of the specimens was annealed for 10 minutes; a second was annealed for the same length of time under an initial creep stress of 15,200 psi, both at 542°C. A third specimen was completely recrystallized by annealing for 20 hours at the same temperature. The fourth specimen was tested in the as cold-rolled state. Room temperature stress-strain curves obtained from the four specimens are shown in Fig. 11. As may be seen from these results, the dynamic-annealed specimen shows by far the most pronounced yield point effect; however, once the dislocations are unpinned the flow stress falls below that of the static-annealed specimen which is consistent with the hardness data. Admittedly, the difference in plastic flow stress here is small.

In order to bring about any rapid interaction between vacancy clusters and dislocations, plastic deformation should be necessary. Several dynamic annealing tests were made at stresses below 7,500 psi. If no plastic strain was observed the hardness coincided with the static annealing data; however,

when plastic strain was obtained the hardness change was seen to fall within the common band shown in Fig. 9. It would thus appear that concurrent plastic straining, and not elastic strain alone, is required during dynamic annealing in order to obtain an additional hardness drop relative to that obtained from static annealing. Although the elimination of vacancy clusters, which will be referred to as declustering, may perhaps account for the initial drop in hardness, it is difficult to explain why this drop should be essentially independent of the amount of concurrent plastic strain caused by stresses up to about 15,200 psi. At stresses of 20,000 and 27,500 psi additional decreases in hardness values are obtained. An examination of the microstructure developed by annealing under a stress of 27,500 psi for 10 minutes at 542^{o}C (Fig. 10G) suggests the presence of polygonization as being responsible for further softening. Additional dynamic annealing at this stress results in the development of a well-defined substructure (Figs. 10H and I). For long-time dynamic annealing at the lower stresses the hardness data may be seen to depart from the common as-annealed hardness band observed at the shorter times (Fig. 9). For example, the data for the 15,200 psi test breaks away after an annealing time of about one hour and approaches a value considerably above the data within the band for the corresponding time. The microstructure developed after 3.6 hours at this stress again shows extensive polygonization. The departure from the common band, the latter being attributed to declustering, may then be ascribed to the presence of polygonization, which in turn is induced by the larger concurrent creep strains. It is well known that the presence of polygonization during the creep of metals, results in the development of a substructure size and of room temperature tensile properties which are dependent upon the creep stress [9,12].

Small concurrent strains during dynamic annealing result in an initial hardness drop without causing any subsequent change in the softening kinetics insofar that polygonization does not take place. It may well be that the

initiation of any polygonization with additional straining at the lower stresses is eliminated by the recrystallization process, at least until the data depart from the common band running parallel to the static annealing curve (Fig. 9). Thus, at the lower concurrent stresses this departure occurs following recrystallization.

The effect of dynamic annealing on the softening kinetics at 481°C, in a region where only recovery takes place during static annealing, is shown in Fig. 12. The results show that a parallelism exists between the dynamic and static annealing data during the early annealing period. Again, an initial rapid drop in hardness below that obtained during static annealing is suggested. The subsequent divergence of the two curves may be ascribed, as for the curves in the above, to the tendency of the material, when under a creep load, to approach a structure and hardness consistent with the state of stress. The development of a substructure under dynamic annealing at 481°C may be seen in the sequence of photomicrographs shown in Figs. 13D-F, in contrast to those obtained under static annealing (BandC).

Since polygonization would appear to be responsible for the differences in softening behavior observed between the static-annealed and dynamic-annealed specimens (following the initial declustering in the latter case), dynamic annealing in a range where polygonization is evident under static annealing conditions was next studied. The effects of dynamic annealing at 510°C under initial creep stresses of 15,200 and 30,000 psi are shown in Fig. 14. The curves shown for both stresses are similar to the dynamic-annealed one obtained at 481°C (Fig. 12) where only recovery took place on static annealing. Fractured specimens only were examined from the 510°C dynamic annealing tests. These showed well-defined substructures, within the originally cold-rolled grains, as a result of creep. The data for both curves in the early annealing period appear to converge within a common band; the latter is shown as dashed lines. This again supports the

interpretation of the sudden drop in hardness below that obtained for the static annealing data; the drop is independent of the amount of concurrent straining insofar that the straining is low, of the order of about less than 3% strain. Under small concurrent strains subsequent softening follows parallel to that of static annealing. At greater strains, polygonization sets in and the data departs from the common band, as was obtained at 481 and 542^{o}C.

It is of interest to note that the data from the 15,200 psi tests converge with the lower range of the static annealing scatter band over part of the annealing period (Fig. 14). This suggests that the softening behaviors for the two types of tests over this range may be similar. Since the additional softening in the dynamic test is attributable to polygonization the lower boundary, X-X, of the static-annealing scatter-band data may also be considered to result from the presence of this softening process. The subsequent falling away of the dynamic data from the static annealing data would be due to the increased acceleration of polygonization as a result of the increasing amount of concurrent strain with time. On the other hand, within the static annealing scatter band competition between the polygonization and recrystallization kinetics is taking place; it would be expected that the exact annealed state at any time may be highly sensitive to slight differences in the initially 50% cold-rolled specimens. Furthermore, when recrystallization does take place here the recrystallization kinetics may be altered due to the reduction in the state of strain brought about by the previous polygonization. This, in effect, would shift the softening points where some recrystallization does eventually take place to times corresponding to a lower initial cold-worked state and in the extreme correspond to the upper boundary Y-Y. The competition between recrystallization and polygonization will be dealt with in more detail in a later section. Where concurrent straining takes place, however, the accelerated polygonization would be sufficient to prevent any recrystallization and thus should correspond to the other extreme, i.e., the lower boundary X-X.

When dynamic annealing is undertaken in a region where polygonization normally accompanies static annealing the softening kinetics, although somewhat speeded up, are closely related, insofar that creep is not excessive. When extensive creep straining takes place the effect is, as in the case of recovery, to more rapidly accelerate the softening; however, the general shape of the softening curve is not significantly altered as was observed for the recovery and recrystallization regions.

C. Dynamic Annealing Followed by Static Annealing

The dynamic annealing results were seen to fall into two areas. First, on the initial application of a load a sudden softening took place as a result of the induced concurrent straining. This was not accompanied by any apparent microstructural change and was independent of the stress insofar that the induced strains were small. The softening was attributed to vacancy declustering. Second, at higher concurrent strains the softening phenomenon was accounted for largely by polygonization. If the distinction between declustering and polygonization be real, then it would be expected that different softening kinetics would be obtained during the static annealing of specimens which have previously been subjected to initial treatments yielding these two conditions, i.e., one in which only vacancy declustering has taken place, and one in which polygonization has been initiated. Prior dynamic annealing under stresses of 15,200 psi and 27,500 psi for 10 minutes at 542°C would yield these respective states, as determined microscopically (see Figs. 10D and G). The effects of such prestrain annealing treatments on the subsequent static annealing hardness data compared with the complete static annealing curve at 542°C may be seen in Fig. 15. Microstructures after 0.8, 1.8 and 30.8 hours at 542°C for the three corresponding curves are shown in Fig. 16.

It is quite evident that no significant differnece in the microstructure exists between the completely static-annealed specimens (A-C) and those obtained

from an anneal following prior dynamic annealing under a stress of 15,200 psi (D-F). With respect to the corresponding hardness data (Fig. 15) the initial hardness spread is more or less maintained until recrystallization is almost completed. A gradual convergence of the two curves then takes place. The data in the early annealing period following the 15,200 psi dynamic anneal (Fig. 15) may be seen to correspond to the band obtained for continual dynamic annealing under stresses ranging from 7,500 to 15,200 psi (Fig. 9). This is again consistent with our interpretation, that the important difference between the static and dynamic annealing curves in this region occurs primarily from phenomena taking place during the period of initial concurrent straining during dynamic annealing and which we have ascribed to declustering.

The results again imply that the elimination of vacancy clusters do not affect the rate of recrystallization. The difference in hardness between the two curves (Fig. 15) throughout the annealing period may be assumed to be due largely to differences in vacancy concentrations. The convergence of the curves suggests that the process of recrystallization must either reduce to an equal concentration or wipe out the vacancy clusters originally present in the cold-worked metal. The difference in levels of the two curves considered indicates that vacancy-cluster hardening represents about 10% of the total increase in strength due to cold work. An analogy to the elimination of vacancy clusters by recrystallization may be seen in the work reported by Robbins who showed that 'micropores' can be eliminated by motion of grain boundaries in sintered copper sheet(26); Cizeron has shown similar effects in the sintering of pure iron(27).

The annealing kinetics (Fig. 15) are modified when preceded by prior dynamic annealing under a stress of 27,500 psi. This stress results in a drop of hardness below that of the common band (Fig. 9) which was ascribed to declustering. The prior polygonization associated with the higher stress resulted in slowing up the subsequent static recrystallization process as indicated by the

hardness data (Fig. 15) and microstructures (Fig. 16). A smaller degree of recrystallization may be seen after a given annealing period for the prior dynamic-annealed specimens (G-I) when compared to those which were not prestrained (A-C). The appearance of the early stages in polygonization seen as a result of the prestrain anneal (G) gradually becomes less evident with subsequent static annealing (G-I). The microstructures and the modification of the softening kinetics would suggest that the presence of the initiation of polygonization subsequently interferes with the recrystallization process; however additional polygonization does not take place on further annealing at this temperature on removal of the concurrent stress. Furthermore, this additional annealing tends to remove the evidence of prior polygonization as the material recrystallizes, although some subboundaries are still apparent near the end of the recrystallization process (Fig. 16I). One may consider the displacement of the softening kinetics to longer times during recrystallization (Fig. 15), again, as resulting from a reduction in the residual strain energy, in both potential nucleation sites and surrounding matrix, caused by the polygonization induced by concurrent prestraining during prior dynamic annealing.

These results indicate that either a critical amount of polygonization is necessary in order to modify the recrystallization kinetics, or that, indeed, two separate phenomena occur during concurrent straining, one of which, declustering, although causing some softening, plays a minor role, if any, in influencing the recrystallization behavior, per se. It was suggested earlier that the yield point effect observed (Fig. 11) was due to the interaction between dislocations and residual vacancies of the clusters. Now, recovery occurs essentially by movement of dislocations along slip planes resulting in either mutual annihilation of unlike dislocations or in the formation of more stable horizontal dislocation arrays. If the dislocations are pinned by vacancies, as a result of a prestrain anneal, then subsequent static annealing in the recovery region should result in

a slowing-up of the softening kinetics as compared to the corresponding complete static annealing curve.

Specimens, first dynamic annealed at 542°C for 10 minutes at a stress of 15,200 psi, were then subjected to static annealing at 481°C. The hardness results are shown in Fig. 17, in which the complete static annealing curve is included. The hardness values of the prestrain-annealed specimens may be seen to remain virtually constant for a subsequent static annealing period of over 818 hours. The data for the two curves appear to converge at about 2740 hours. Microstructures of specimens at a magnification of 2000 representing both curves are shown in Fig. 18. No significant difference exists between the as-prestrain-annealed and subsequently 818-hour-annealed specimens (D and E). The only visible change after 2740 hours (F) is the presence of some small new grains. The new grains and the appearance of the annealing curves at this time (Fig. 17) suggest the initiation of recrystallization. The microstructure observations for the completely static-annealed specimens (A-C) are very similar to those made on specimens initially in the prestrain-annealed condition. Furthermore, no significant difference exists between the two sets of microstructures for the same corresponding times. A small amount of lineal structure is suggested as being present in all specimens, perhaps indicative of the initiation of a substructure. This appears to be more readily visible in the prestrained specimens; however, the amount of such a structure seems to remain unchanged with annealing for both series of specimens (A-C and D-F). It would appear that this lineal structure, if significant, is developed soon after reaching temperature. Thus, it may be argued that this structure is associated with the phenomena responsible for the sudden change in hardness from the cold-worked state (Fig. 6).

The important consideration here, however, is the observation that no significant drop in hardness takes place during at least the first 818 hours of annealing following a prestrain treatment. This again supports our contention

that some interaction between vacancies and moving dislocations may have taken place during the prestrain anneal. Perhaps some initial polygonization occurs with declustering. The interaction between the residual vacancies and moving dislocations prevents further subboundary formation and growth; accordingly, the microstructure does not change with additional annealing until a new process, recrystallization takes over. One must then assume, however, that a similar phenomen takes place without a prestrain anneal, for a lineal structure, although to a lesser extent, is also observed here. This phenomenon, then must be independent at least to some degree, of the strain-induced declustering. Perhaps the prestrain anneal, along with causing declustering, accelerated the development of the lineal structure consistent with the effect of concurrent straining on polygonization. The subsequent movement of dislocations, as necessary for further subboundary development, is impeded by vacancy-dislocation interaction in probably both the prestrain-annealed and unprestrained specimens of the 50% cold-rolled condition, the difference being essentially one of degree brought about by the additional effect of concurrent straining. It is of interest to note that the hardness values for both curves are the same at the time corresponding to the onset of recrystallization. One might then assume that potential recovery dislocations were those which interacted with the residual vacancies on declustering during the prestrain anneal, and also took part in the formation of the lineal structure.

These annealing tests were repeated for specimens prestrained at the same test temperature of 481°C. Both the hardness behavior and microstructure obser-varions were similar to what were described in the above for a prestrain temperature of 542°C.

GENERAL DISCUSSION ON RECOVERY, POLYGONIZATION, AND RECRYSTALLIZATION

A. Softening by Recovery

Recovery phenomena, in which no interfacial movement is involved, are generally depicted as occurring almost linearly with logrithmic time after an initial rapid change in the cold-worked state[28,29]. The initial rapid change is consistent with the microstructure observations (Figs. 4-6) described in a previous section. It was suggested that an almost instantaneous movement of the more easily movable dislocations away from the more highly strained regions takes place. Such regions would be at severely bent glide planes (deformation bands). It would appear that this rapid phenomenon is not thermally activated.

An explanation for the observed early recovery behavior may be that at the elevated temperature the critical shear stress for yielding in the cold-worked metal is reduced to a value below that of the residual back stresses acting on the piled-up dislocations. Then, on reaching temperature, one would expect an immediate sudden back-movement of the dislocations associated with the higher residual stress fields. Recovery, thereafter, will occur primarily by thermal activation of single or very small groups of dislocations along glide planes resulting in a more stable configuration or in mutual annihilation. This process will slow down with time (indicated by the almost logrithmic linearity) as the more easily movable dislocations have readjusted their position until another process, such as polygonization or recrystallization, takes over.

Since the recovery processes as depicted here do not involve the formation of a new interface nor the presence of nucleation and growth phenomena as indicated by the absence of an incubation period, the relative rates of recovery should be independent of the degree of prior cold work. The relative decrease in hardness from four different cold-worked states as a function of annealing time at 481°C is shown in Fig. 19. The coincidence of the data are in agreement with

the above explanation; the separation of the annealing data of any cold-worked state from the common curve occurs when a new process intervenes.

B. Softening by Polygonization

The phenomenon of polygonization together with the effect of material and test variables on its behavior have been described in several excellent articles [4,6-11]. In general, the softening by polygonization during the annealing of a cold-worked metal occurs more readily both the purer the metal and the less complex the deformation. With increasing temperatures, the removal of the cold-worked state is more likely to occur by recrystallization. Polygonization, which is a thermally activated process, takes place by the combination of movement of excess edge dislocations of one type along glide planes and climb parallel to these planes lining up so as to form low-angle boundaries. These boundaries separate slightly disoriented regions which are referred to as substructures. The boundaries may subsequently move and coalesce resulting in a growth of these substructures. With additional time the boundaries may merge with the originally cold-worked grain boundary.

Because of both the impurity content and the complex dislocation pattern probably obtained in the cold rolling of a polycrystalline metal, as existed in our case, polygonization was found to occur in our investigation only over a very limited temperature-time range (Figs. 2 and 3). This limited range may be partially explained with reference to the schematic softening curves shown in Fig. 20, in which the solid lines refer to recrystallization alone starting from the value H_0. H_R refers to the cold-rolled hardness; the difference, H_0-H_R, is due to some softening mechanism other than recrystallization, e.g., recovery. The dashed lines refer to polygonization. The implication here is that polygonization is an easier (though slower) process and has a lower activation energy. At a high temperature, T_1, no interference by polygonization is encountered and the softening proceeds completely by recrystallization. At a lower temperature,

T_2, polygonization may have advanced to such an extent in many of the grains that the residual strain energy remaining is insufficient for the potential growth of high-angle boundary nuclei, or the formation of such nuclei is delayed to a later time as indicated by the solid circles. Thus a mixed polygonized and recrystallized structure is obtained due to the competition of these two softening mechanisms. This concept is consistent with our results as verified by the microstructures shown in Figs. 7B and C and 8F. The kinetics in such a region should be very sensitive to minor variations from specimen to specimen resulting in an erratic type of behavior and considerable scatter in hardness. Presumably one should obtain a completely polygonized structure at some temperature below T_2.

Schematic curves, similar to those shown in Fig. 20, could be drawn comparing the recovery and polygonization kinetics whereby below a certain temperature recovery should predominate. Although we have emphasized differentiating between polygonization and classical recovery, which we feel is justified on the basis of relative changes in mechanical properties and microstructures which are obtained, one must keep in mind that many similarities do exist. Neither require nucleation (i.e. no incubation period), both involve thermal activation of dislocations along glide planes and climb perpendicular to these planes. In the case of recovery, climb involves relatively short distances, the climb being motivated by dislocations of opposite sign generally resulting in mutual annihilation. In contrast, in polygonization, climb would involve relatively larger distances involving dislocations of like sign which line up vertically. It would appear that in the very early stages, both processes would be essentially identical and indistinguishable, and consequently, it would be rather surprising if at least some indication of the initiation of polygonization was not present with recovery.

The presence of polygonization at the annealing temperature of 481°C has been suggested and discussed with reference to the photomicrographs shown in Fig. 16, in which a faint scattered lineal structure may be seen for the recovered as well

as for the prestrain-annealed and subsequently recovered specimens. The question of some very early stage in polygonization taking place here was consequently brought up. A very strong point, however, in favoring the minor role played by polygonization at this temperature is the lack of change in the microstructure with time in contrast to the changes observed at 510°C during annealing.

The end of the softening curve for the 50% cold-rolled state at 481°C, shown in Fig. 2, appears to be headed into a region of rapid hardness drop at a hardness value corresponding to that at the higher temperatures, suggesting the initiation of recrystallization. In fact, Fig. 18C shows evidence of some recrystallization after an annealing period of 2740 hours; in contrast, however, there are no visible signs indicating that (continued) polygonization has taken place following the initial formation of the lineal structure. Perhaps at this temperature the recovery process per se interferes with polygonization at a very early stage in its development. It was suggested in an earlier section that such interference may take place as a result of interaction of dislocations with single vacancies or vacancy clusters; further softening by polygonization is then prevented. Thus, for the 50% cold-rolled condition polygonization, which is shown to become predominant for an annealing temperature of 510°C, contributes a negligible amount, if at all, to softening at 481°C. If a temperature range does exist for the complete elimination of the strained state by polygonization alone for this cold-rolled condition, then this range must indeed be small (at some temperature between 481 and 510°C).

Although polygonization plays a minor and limited role during static annealing, apparently being effective only over a narrow temperature range, it assumes an important role at all annealing temperatures studied if concurrent straining is involved, but only above some small critical induced strain. The effect of concurrent straining during annealing would be to shift the dashed curves of Fig. 20 to lower times without affecting the solid lines, as concurrent

straining does not appear to affect recrystallization per se. The effect here is indirect, either interrupting the process or altering it by modifying the strain energy state of potential nuclei and growth sites. Here, the straining would add to the frequency factor in dislocation movement during polygonization. This is consistent with the widely accepted concept that high temperature deformation proceeds by dislocation climb and glide[31,32]. The result of this shift is to interrupt more effectively the recrystallization process over a wider range of temperatures. In the recovery region polygonization is induced where it was previously difficult to observe. Since concurrent straining under dynamic annealing conditions favors polygonization, the effect of this straining in the so-called classical recovery region is to accelerate greatly any polygonization which may have taken place, although undetected, and thus, in a sense, separate the two phenomena as interpreted from the static and dynamic annealing curves. In the final analysis one must bear in mind that under larger concurrent strains the processes become more and more complicated due to the phenomenon of creep, per se.

C. Softening by Recrystallization

Several excellent review articles have been written in recent years summarizing the work done in the field of recrystallization[3,4]. In brief, the process is depicted classically as occurring by the growth of strain-free nuclei into the surrounding deformed material, the driving force being the difference in volume energy existing between the soft and deformed states across the moving interface. Disagreement exists as to whether these strain-free nuclei form directly in the deformation process or whether they form after reaching temperature. It would appear, in view of an incubation time involved, that the latter case is more consistent with experimental observations. Furthermore, this incubation period is dependent upon strain which should not be the case if 'preformed' stable nuclei already existed.

Cahn has suggested that the recrystallization nuclei form by a process of polygonization[33]. Recently, Bollman, with the aid of electron microscopy techniques, described the similarities and differences in the mechanism involved between polygonization and recrystallization[34]. Although both processes are somewhat similar, in that they depend on the movement of dislocations, they develop independently. Recrystallization will be favored in those regions where considerable misfit in crystal orientation, such as present at deformation bands or grain boundaries, exist. A nucleus forms by volume dislocations moving out of a strained region into a boundary, which is already a high-density dislocation boundary separating those regions differing considerably in orientation. This strain-free nucleus then grows by the movement of the boundary into the strained region, the latter acting as a sink for the volume dislocations responsible for the residual state of strain. The driving force necessary for the movement results from the difference in volume (strain) energy between the regions on either side of the boundary. Thus both the initiation of the nucleus and its subsequent growth are dependent on the state of strain.

In Fig. 21 the effect of percent cold-reduction on recrystallization, as indicated by the time to reach the start of a rapid drop in hardness, is clearly seen. An increase in cold reduction shifts this time to lower time intervals. This is in contrast to the results shown in Fig. 19, for the recovery region, where the relative softening rate is independent of the amount of prior strain. The kinetics and microstructure observations made in the recrystallization regions here are consistent phenomenologically with the classical concept; the latter is modified only with respect to the atomistic details by the introduction of dislocation movement.

If the recrystallization phenomenon is governed by a single process then the kinetics should be described by a single value of activation energy dependent only on the type of material. We would like to suggest a possible explanation

for the disagreement frequently reported in the literature on the effect of temperature and state of strain on the activation energy for recrystallization. Both the independence and dependence of the activation energy on these variables have been claimed[3]. The activation energy is determined by comparing the kinetics of supposedly identical states at two different temperatures, generally by the measurement of some physical property such as hardness. If polygonization be responsible, in part, for the softening, then identical states may not exist for the same value of the measured property, as the relative contributions by polygonization and recrystallization may be different at the two temperatures; the kinetics, then, are actually not due solely to recrystallization and a fictitious activation energy is obtained. Since other softening mechanisms such as recovery and polygonization are likely to play an increasingly important role at lower deformations and lower strains, it might well be that the dependence of the activation energy on these two variables, as reported in the literature, is actually the result of the measurement of a fictitious value.

Although numerous rate expressions have been developed to explain recrystallization kinetics[3], a good approximation of many of these laws may be expressed in the form of the Arrhenius Equation for a thermally activated process, namely, $r=r_o[\exp(-Q/RT)]$. Here, r=rate, r_o=constant, T=absolute temperature, R= gas constant, and Q=activation energy. Expressing r in the form of dH/dt, where H refers to hardness, the expression $t_H= K[\exp(Q/RT)]$ is obtained, where t_H= time to reach hardness, H, and K=constant. The value of Q/R, and thus the activation energy for the process, may then be obtained from a plot of log t_H versus 1/T. Fig. 22 shows such a plot, using a difference in time for t_H, for the 15, 50 and 75% cold-rolled conditions. In order to reduce the error which may arise in the time of heating and cooling the specimens and to minimize the presence of softening mechanisms other than recrystallization, the time value is taken from the region of rapid hardness drop. Thus, the time represents the difference in time

to reach a given value of hardness just beyond the start of rapid hardness drop and a second common value prior to the levelling off of the hardness for the curves in each of the Figs. 1-3.

With reference to Fig. 22, it may be seen that the points obtained at the higher temperatures lie on straight lines for each of the cold-worked states. Activation energies of 98.9, 93.1 and 85.1 kilocalories per gram mole are obtained for the 15, 50, and 75% cold-rolled conditions, respectively, from the slopes of these lines. The dashed lines A-B, which represent the extension of the straight lines on the assumption of the existance of a constant activation energy, have been displaced by the experimental points to A-A resulting in abnormally high apparent activation energies. The displaced curves are due to a slowing up of the recrystallization process due to the interference of other softening mechanisms. The temperatures for the points along the curves A-A may be seen to correspond to those curves in Figs. 1-3 in which the annealed hardness levelled off at relatively higher values or exhibited considerable scatter, thus, regions where polygonization, particularly in the latter case, was found to play an important role in softening.

If recrystallization is a single-activated process then the activation energy should be independent of both temperature and initial state of strain. The effect of decreasing temperature on this energy has been ascribed to the appearance of other softening mechanisms. The fact that the activation energy increases somewhat with decreasing cold-work may perhaps be explained on the same basis. Since longer incubation periods are required for the lesser cold-worked states, the relative degree to which a non-nucleating process has advanced prior to the onset of recrystallization should be greater. Thus, interference to recrystallization would increase with lesser cold-worked states; the effect would increase with decreasing temperatures and accordingly the slope of the t_H versus 1/T curves would increase. The break from the initial straight line, as indicated by A-A

in Fig. 22, should correspond to where the competing process continues throughout the softening period; presumable, at higher temperatures the recrystallization has completely wiped away any evidence of any other previous softening mechanisms. It is of interest to note that the departure from the initial curves in Fig. 22 takes place at higher temperatures with lower prior cold work, consistent with experimental observations that the ease of other softening mechanisms taking place in preference to recrystallization increases with decreased cold work and thus higher temperatures are necessary for favoring recrystallization.

If the possibility of one of the softening phenomena taking place depends on the existence and degree to which another mechanism has preceded it, then one should be able to explain the effect of purity, degree of prior cold work, the amount of concurrent straining, and temperature on the relative ease of occurrence of these softening mechanisms. Referring to Figs. 19 and 21, since the nucleation-type process is dependent on the existing state of strain the lesser cold-worked state will shift the nucleation-type curves to longer times while essentially not affecting the non-nucleating processes. Thus the likelihood of the latter type of process, such as recovery or polygonization, being prevalent will increase with decreasing amount of prior cold work. Referring, again, to Fig. 21, we have already described how a decrease in temperature will also favor the presence of these latter processes. The effect of concurrent straining on accelerating polygonization has already been discussed in some detail. With respect to purity, there is considerable experimental evidence showing that the polygonization occurs more readily in favor of recrystallization in high purity metals [10,11]. This is consistent with our knowlege of the slowing up of dislocation movement by impurity atoms. Thus, with purer metals, the non-nucleating curves, which are more dependent on purity, would be more readily displaced to shorter times with respect to the recrystallization curves. It would appear that in the investigation of recrystallization, one is less likely to encounter erratic or

apparently anomolous behavior when metals of lesser purity are used; a more accurate picture of the recrystallization kinetics per se, thus, would be obtained.

SUMMARY

The softening kinetics of a cold-rolled low carbon steel when annealed with and without an externally applied stress were studied in some detail. Four important processes appear to control the annealing behavior of the cold-worked state, namely, 1. recovery, where no visible evidence of any continued microstructure change exists, 2. polygonization, manifested by substructure formation and growth, 3. recrystallization, involving formation and growth of high-angle boundary nuclei eventually resulting in complete removal of the originally elongated grains, and 4. declustering, defined by the elimination of vacancy clusters by the impingement of dislocations during dynamic annealing.

The criterion for determining the annealing behavior of a material can be predicted from an understanding of the relative shape of the softening curves and differences in activation energies of the various processes. When the polygonization curve overlaps that for recrystallization, potential recrystallization nucleation sites are consumed or modified and erratic softening kinetics may be observed.

Recovery and recrystallization kinetics are relatively unaffected by the presence of small concurrent strains during annealing. An initial relative displacement of the respective curves is obtained which is attributed to declustering. With additional concurrent strains the annealing curves are significantly modified when compared to those obtained under static annealing. The strain-induced polygonization is responsible for these changes in a region where only recovery or recrystallization normally would occur.

Where polygonization predominates during static annealing the effect of concurrent straining is to augment its presence resulting in a general increase

in softening without any significant change in the shape of the annealing curve. This would be expected to occur for two reasons. First, dislocation motion would be accelerated due to concurrent straining, thus yielding a more likely situation for dislocation rearrangement in the formation of low-angle boundaries, and second new dislocation intersections of the screw-screw type would result in isolated vacancies which would be expected to increase the rate of dislocation climb necessary in the formation of subboundaries. The presence of a concurrent strain will thus favor those conditions accelerating polygonization relative to recovery and/or recrystallization.

A qualitative consideration of the various softening kinetics explained the effect of temperature, prior cold work, and purity of metal in determining which of the competing processes would be dominant.

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REFERENCES

1. J. Czochralski, "Historical Summary of Work on Recrystallization up to 1921", Zeit. f. Metallkunde, Vol. 19, 1927, p. 316.

2. H. F. Kaiser and H. F. Taylor, "The Effect of Type of Cold Deformation on the Recrystallization Properties of Armco Iron", Transactions, ASM, Vol. 27, 1939, p. 227. (Extensive bibliography up to 1934).

3. J. E. Burke and D. Turnbull, "Recrystallization and Grain Growth", Progress in Metal Physics, Vol. 3, 1952, p. 220, Pergamon Press.

4. Paul A. Beck, "Annealing of Cold-Worked Metals". Advances in Physics, Vol. 3, 1954, p. 245.

5. Michael B. Bever, "On the Thermodynamics and Kinetics of Recovery", ASM Seminar on Creep and Recovery, 1957, p. 14.

6. R. W. Cahn, "Internal Strains and Recrystallization", Progress in Metal Physics, Vol. 2, 1950, p. 151. Pergamon Press.

7. A. Guinier and F. Tennevin, "Researches on the Polygonization of Metals", Progress in Metal Physics, Vol. 2, 1950, p. 177. Pergamon Press.

8. W. R. Hibbard, Jr., and C. G. Dunn, "Polygonization", ASM Seminar on Creep and Recovery, 1957, p. 52.

9. E. C. W. Perryman, "Recovery of Mechanical Properties", ASM Seminar on Creep and Recovery, 1957, p. 111.

10. M. Jean Talbot, "Etude de la Polygonisation du fer de Zone Fondue; Competition Entre la Polygonisation et la Recristallisation", Report, International Colloquium on the Properties of High Purity Metals, Oct. 12-14, 1959, Paris, France.

11. Jean Montuelle, "Etude de la Polygonisation de P'Aluminum de Zone Fondue; Competition Entre ce Phenomene et la Recristallisation", Report, International Colloquium on the Properties of High Purity Metals, Oct. 12-14, 1959, Paris, France.

12. Oleg D. Sherby, Alfred Goldberg and John E. Dorn, "Effect of Prestrain Histories on the Creep and Tensile Properties of Aluminum", Transactions, ASM, Vol. 46, 1954, p. 681.

13. A. Hultgren, A. Josefsson, E. Kula and G. Lagerberg, "Veining in Ferrite", Jl. of Iron and Steel Inst., Vol. 188, 1958, p. 247.

14. W. A. Wood and G. R. Wilms, "Mechanism of Creep of Metals". Jl. Inst. of Met., Vol. 75, 1949, p. 693.

15. Nicholas J. Grant and Arup R. Chaudhuri, "Creep and Rupture", ASM Seminar on Creep and Recovery, 1957, p. 284.

16. P. Gay, P.B. Hirsch, and A. Kelly, "X-Ray Studies of Polycrystalline Metals Deformed by Rolling: III, The Physical Interpretation of the Experimental Results", Acta Crystal, Vol. 7, 1954, p. 41.

17. Symposium on "Vacancies and Other Point Defects in Metals and Alloys", Institute of Metals Monograph No. 23, 1957, London.

18. R.E. Smallman, K.H. Westmacott, and J.H. Coiley, "Clustered Vacancy Defects in Some Face-Centered Cubic Metals and Alloys". Jl. Inst. of Met., Vol. 88, 1959, p.127.

19. Frederick Seitz, "On the Generation of Vacancies by Moving Dislocations", Advances in Physics, Vol. 1, 1952, p.43.

20. P. Coulomb and J. Fridel, "On the Formation of Cavities Along Dislocations", Dislocation and Mechanical Properties of Crystals, Lake Placid Conference, 1957, p.555, John Wiley & Sons.

21. R. Maddin and A.H. Cottrell, "Quench Hardening in Aluminum Single Crystals", Philosophical Magazine, Vol. 46, 1955. p.735.

22. F. Rohner, "A Theory of the Age Hardening of Aluminum-Copper Alloys Based on Vacant Lattice Sites", Jl. Inst. of Met., Vol. 73, 1947, p.285.

23. H. Kimura, R. Maddin, and D. Kuhlmann-Wilsdorf, "Quenched-In Vacancies In Noble Metals - II, Mechanism of Quench Hardening", Acta Met., Vol. 7, 1959, p.154.

24. M. Wintenberger, "Study of the Elimination of Defects in Pure Aluminum by Means of Low Temperature Resistivity Measurements", Report, International Colloquium on the Properties of High Purity Metals, Oct. 12-14, 1959, Paris, France.

25. A.H. Cottrell, "Theory of Dislocations", Progress in Metal Physics, Vol. 1, 1949, p.77. Interscience Publishers, Inc.

26. D.A. Robbins, Discussion, P.53, Plansee Proceedings 1958. Third Plansee Seminar, "High-Melting Metals", Published by Metallwerk Plansee AG., Reutte, Tyrol, Austria, 1959.

27. G. Cizeron and P. La Combe, "Influence des Chauffages de Part et d'Autre du Point de Transformation Alpha-Gamma du Fer Sur les Processes d'Autodifussion dans le Frittage", Rev. de Metal, Vol. 53, 1956, p.819.

28. P. Gordon, "Microcalorimetric Investigation of Recrystallization of Copper", Transactions AIME, Vol.203, 1955, p.1043.

29. G. Borelius, S. Berglund and S. Sjoberg, "Measurements on the Evolution of Heat During the Recovery of Cold-Worked Metals", Arkiv f. Fysik, Vol. 6, 1952, p.143.

30. R. Drouard, J. Washburn and Earl R. Parker, "Recovery in Single Crystals of Zinc", Transactions, AIME, Vol. 197, 1953, p.1226.

31. J. Weertman, "Theory of Steady-State Creep Based on Dislocation Climb", Jl. Appl. Phys., Vol. 26, 1955, p. 1213.

32. N. F. Mott, "Dislocations, Plastic Flow, and Creep", Proceedings, Royal Society, Vol. 220A, 1953, p. 1.

33. R. W. Cahn, "A New Theory of Recrystallization Nuclei", Proceedings Phys. Soc., Vol. 63A, 1950, p. 323.

34. W. Bollmann, "Electron-Microscopic Observations on the Recrystallization of Nickel", Jl. Inst. Met., Vol. 87, 1959, p. 439.

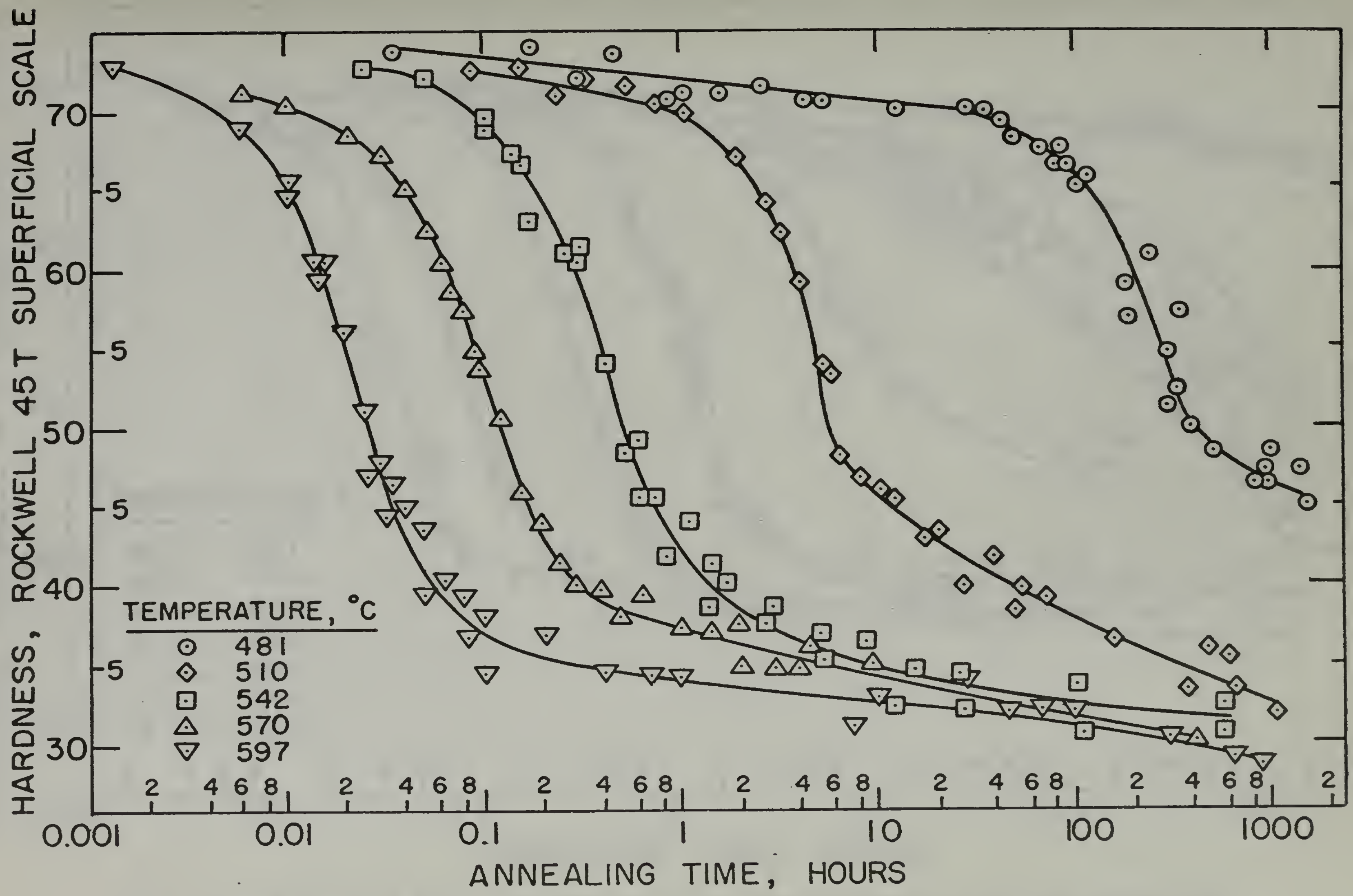
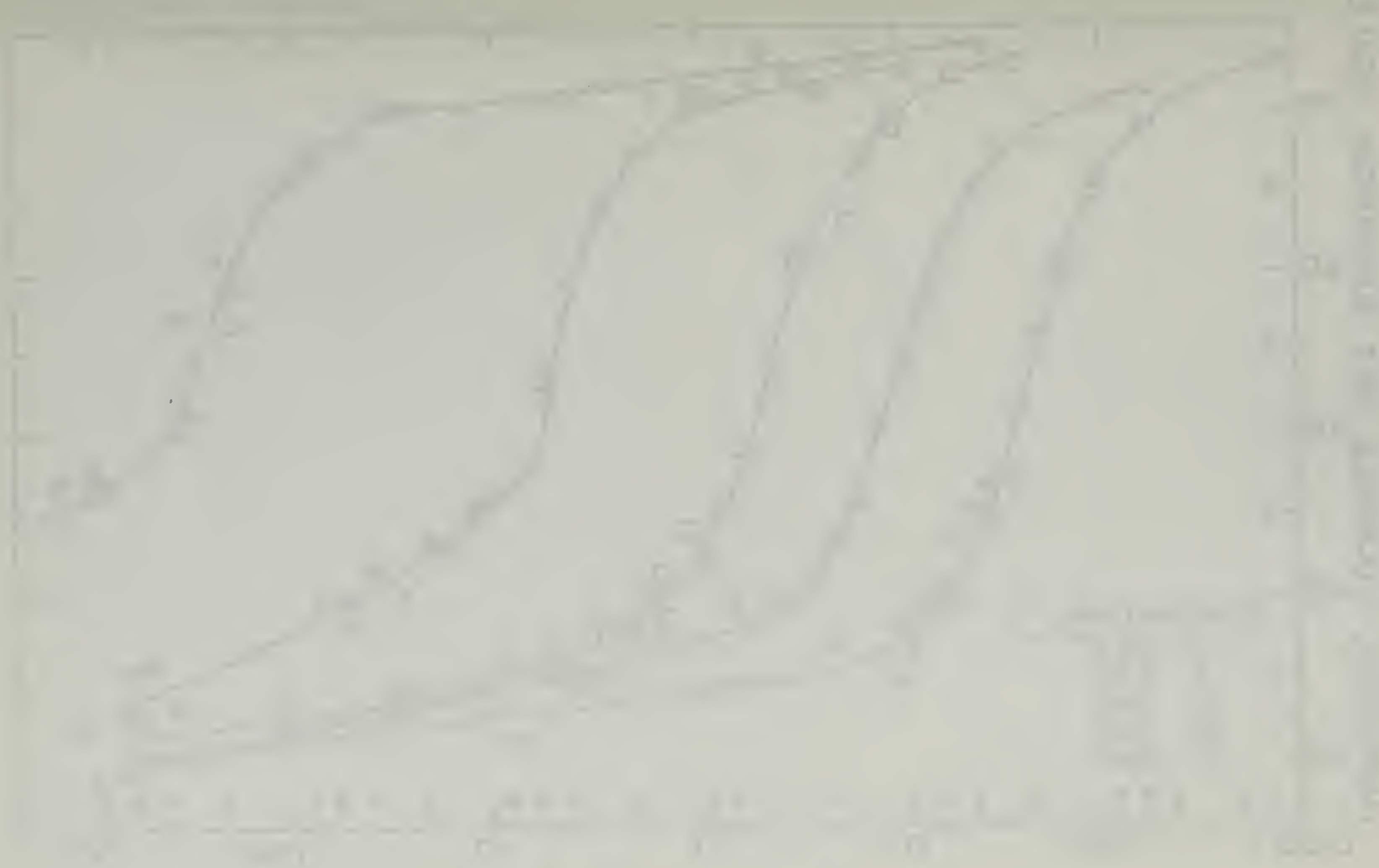


Fig. 1 The effect of annealing time at various temperatures on the room temperature hardness for a 75% cold-rolled low carbon steel.



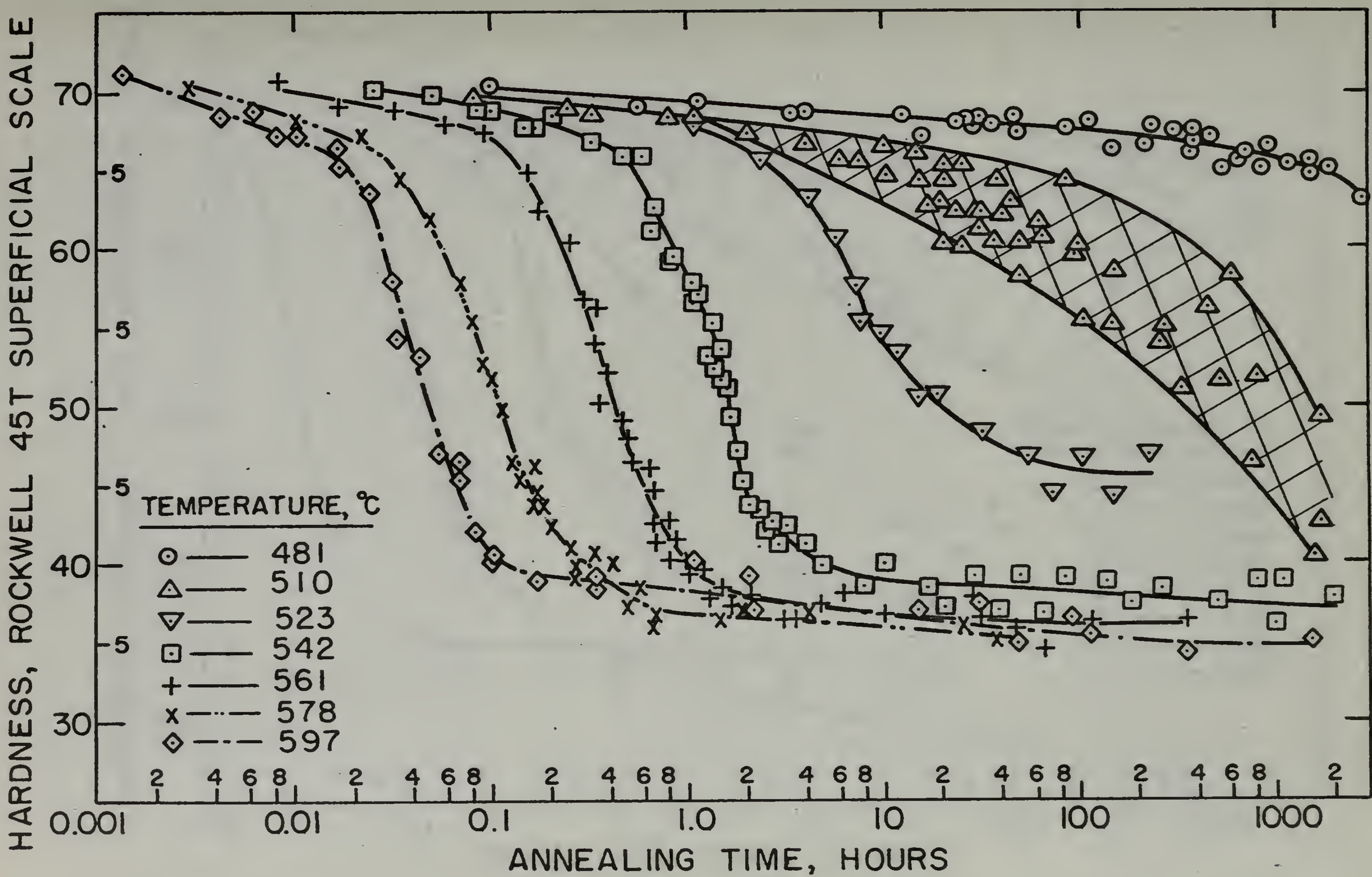


Fig. 2 The effect of annealing time at various temperatures on the room temperature hardness for a 50% cold-rolled low carbon steel.

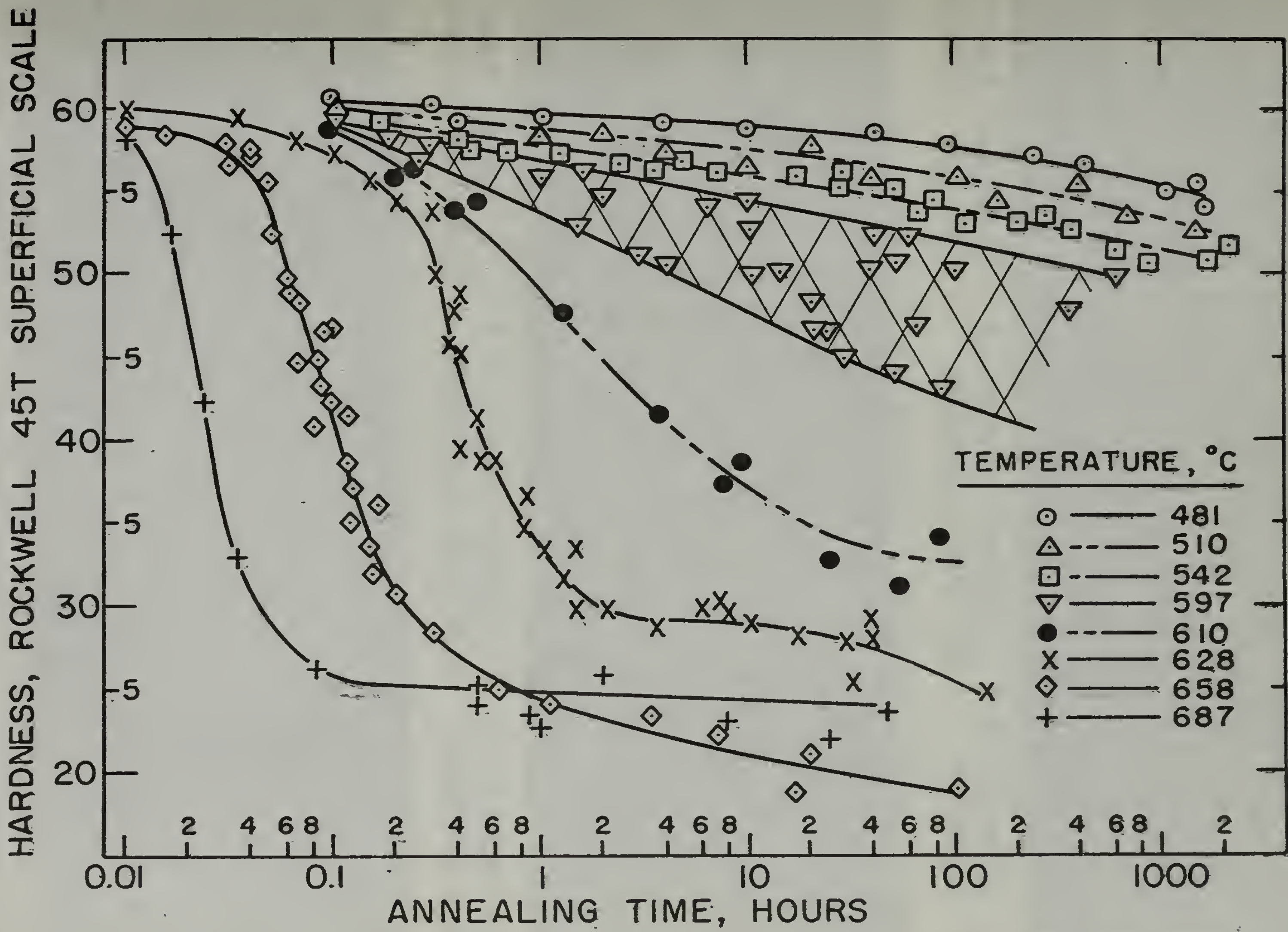


Fig. 3 The effect of annealing time at various temperatures on the room temperature hardness for a 15% cold-rolled low carbon steel.

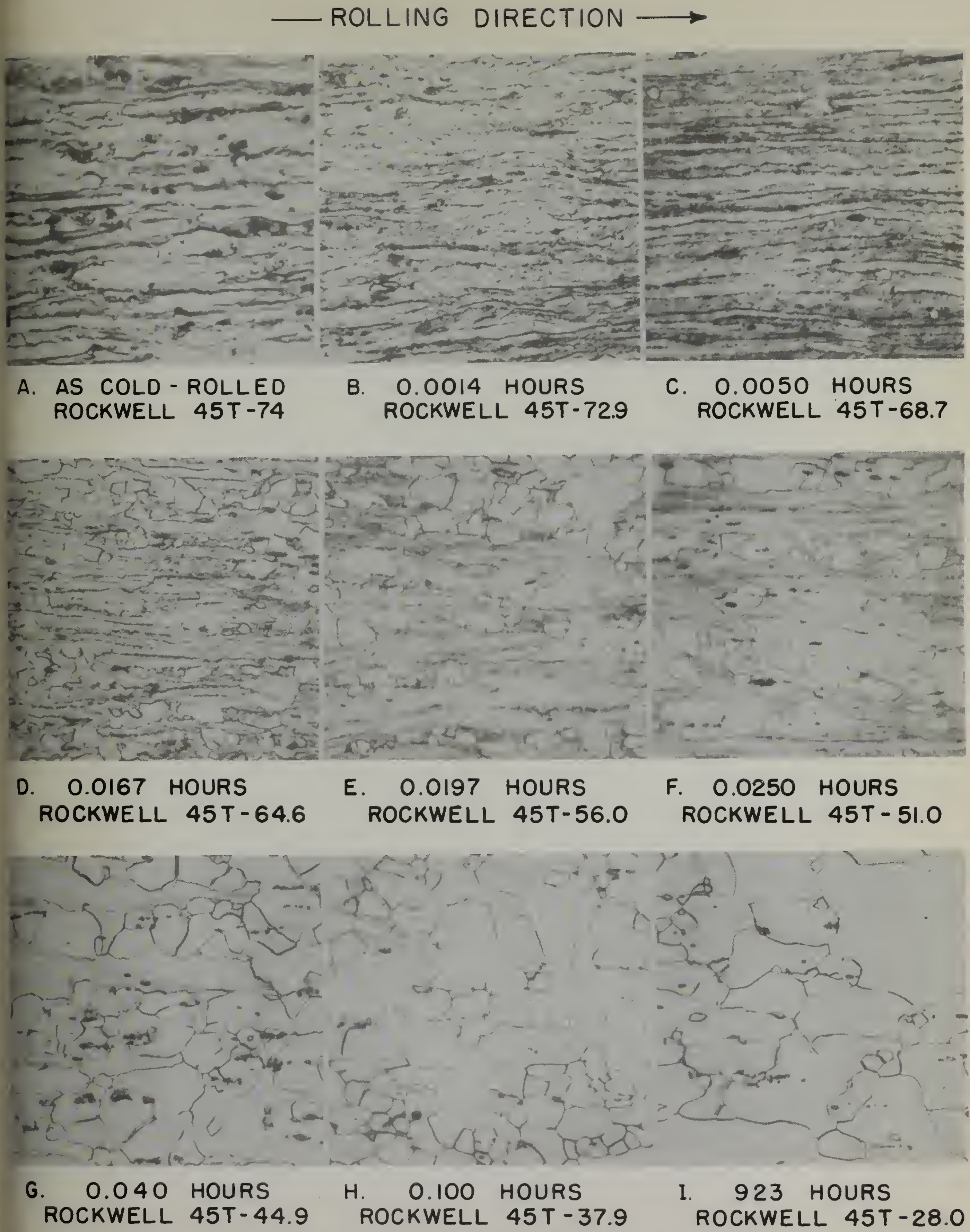


Fig.4 Photomicrographs depicting the parallel change in microstructure with room temperature hardness for a 75% cold-rolled low carbon steel annealed at 597°C, where recrystallization predominates. (Hours refer to time at temperature. X750)

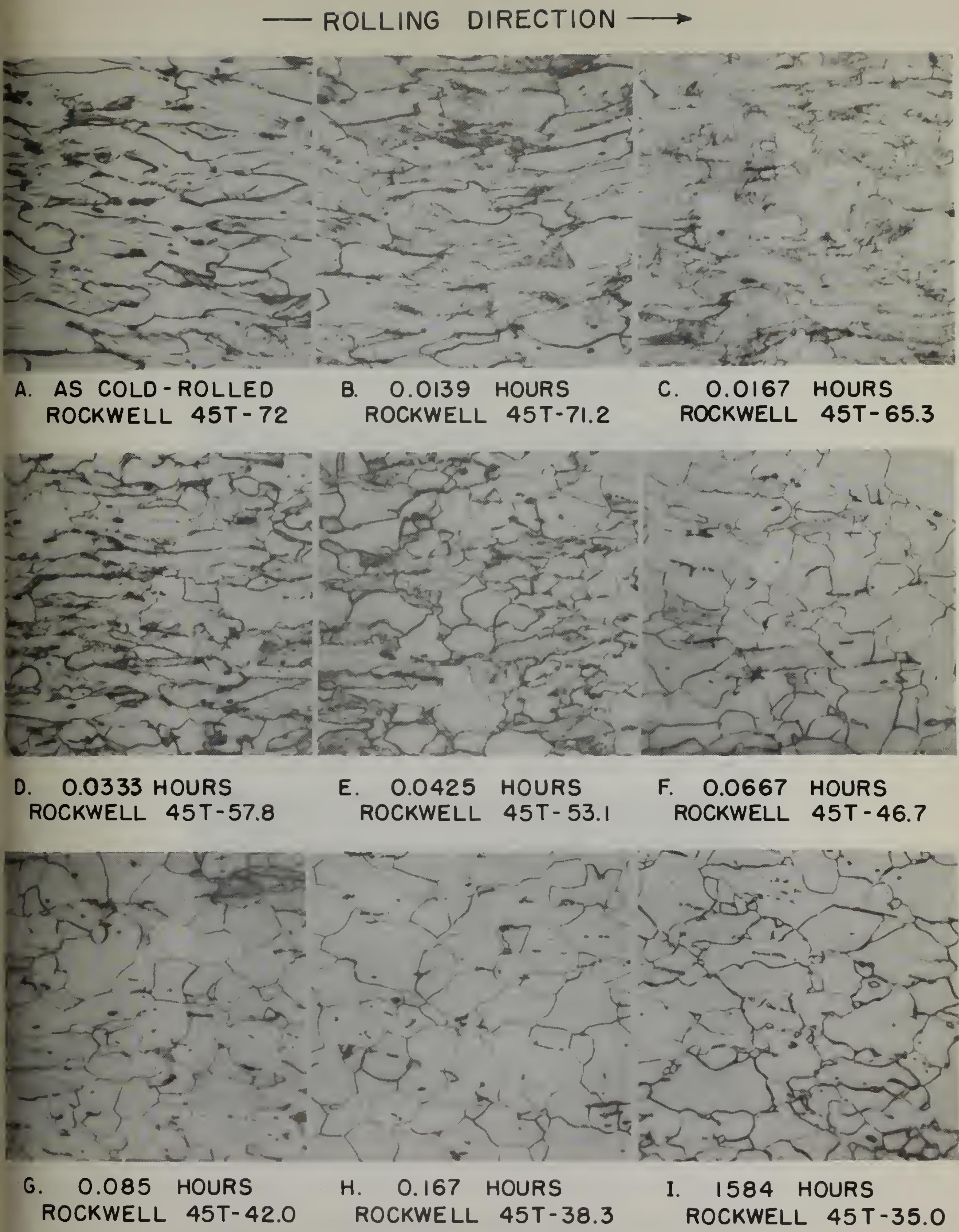
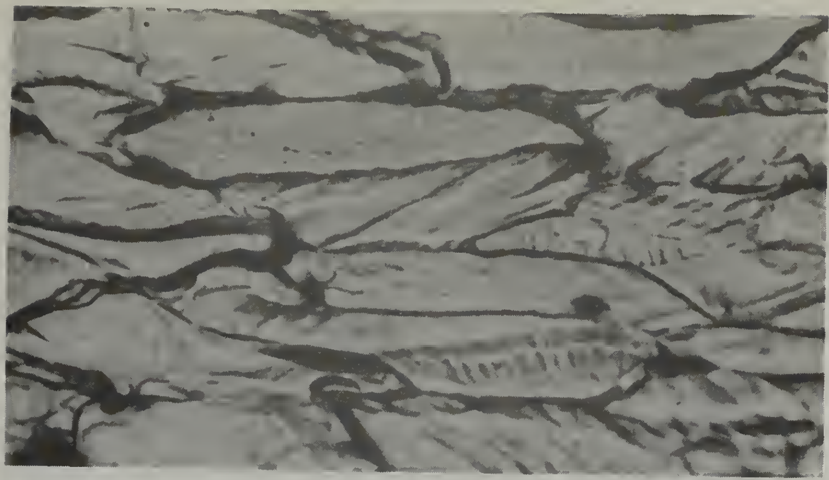


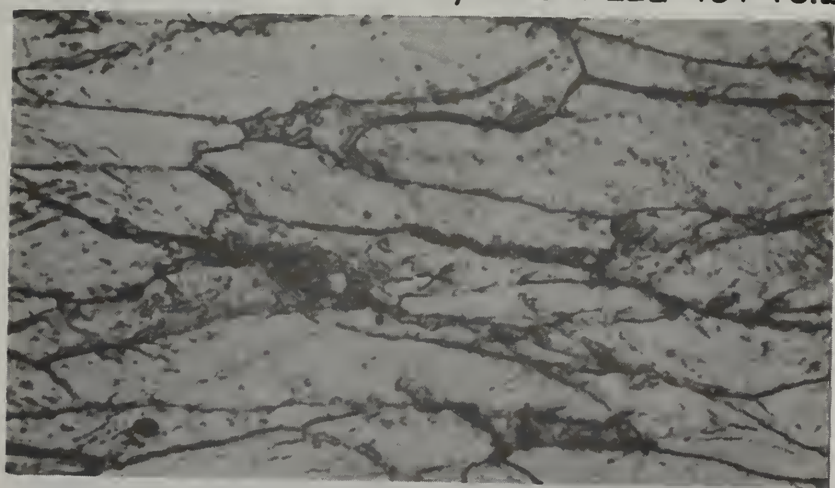
Fig.5 Photomicrographs depicting the parallel change in microstructure with room temperature hardness for a 50% cold-rolled low carbon steel annealed at 597°C, where recrystallization predominates. (Hours refer to time at temperature. X750)

—— ROLLING DIRECTION ——→



A. AS 50 % COLD-ROLLED, ROCKWELL 45T-72

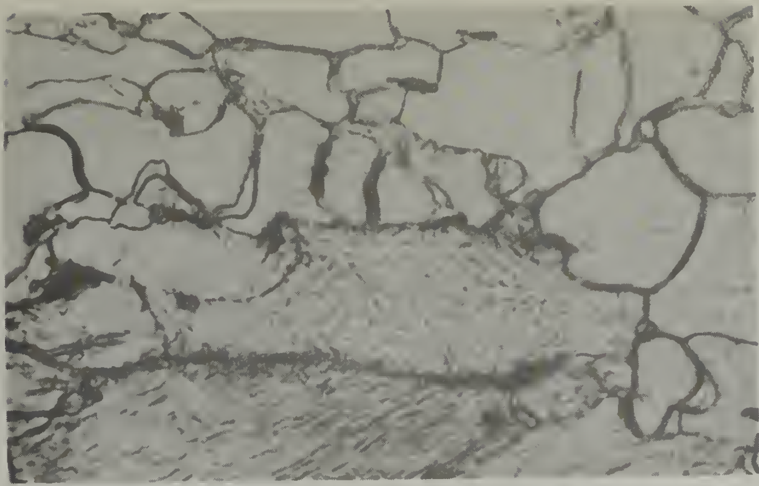
B. 0.10 HOURS AT 481 °C, ROCKWELL 45T-70.2



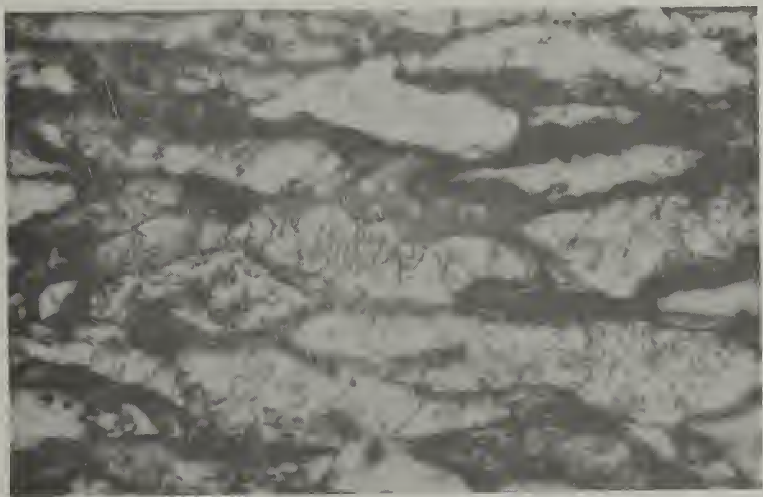
C. 818 HOURS AT 481 °C, ROCKWELL 45T-65.1

Fig.6 Photomicrographs showing the 50% as cold-rolled state, the change in microstructure following an early anneal, and the absence of any further change taking place on additional annealing in the recovery region at 481°C. (X2000)

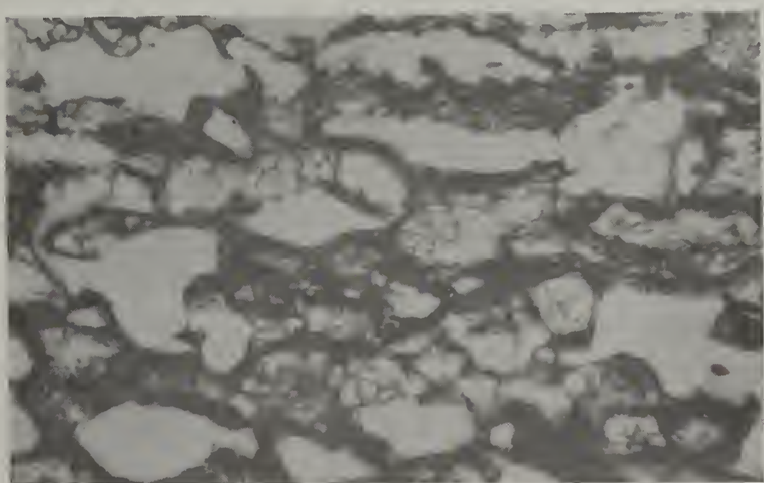
——ROLLING DIRECTION——→



A. 0.0425 HOURS AT 597 °C, ROCKWELL 45T-53.1



B. 349.5 HOURS AT 510 °C, ROCKWELL 45T-51.4



C. DIFFERENT AREA OF SAME SPECIMEN AS B.

Fig.7 Microstructures of two specimens, initially 50% cold rolled, annealed at two temperatures to approximately the same hardness, depicting recovery, recrystallization, and polygonization. (X2000)

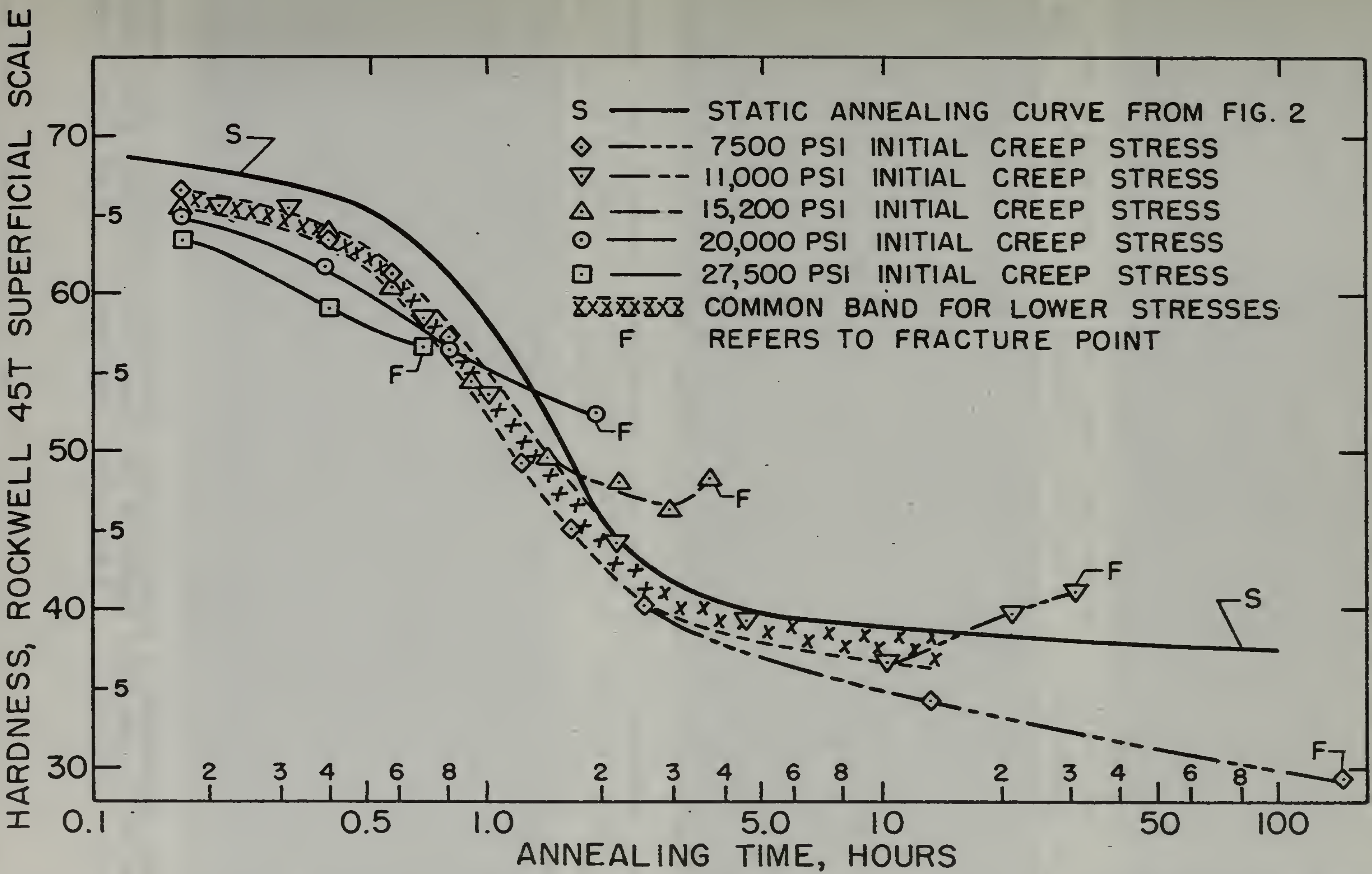


Fig. 9 The effect of dynamic annealing on the hardness of a 50% cold-rolled low carbon steel, as compared to static annealing, in the range where recrystallization predominates at 542°C. Hardness data are obtained at room temperature. Creep tests are interrupted for hardness measurements. Load is applied throughout annealing period for dynamic annealing tests.

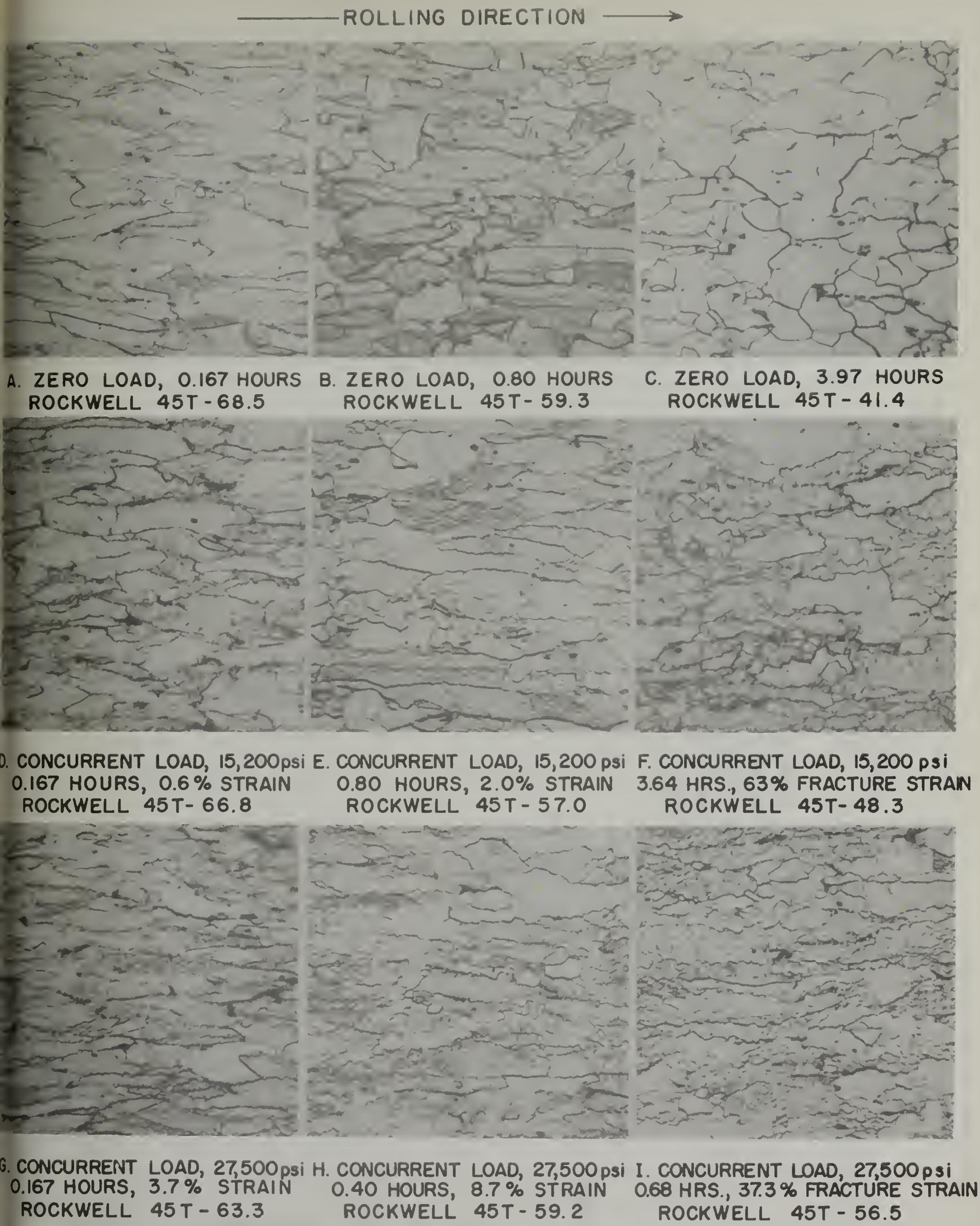


Fig.10 Photomicrographs showing relative changes in microstructure, with annealing
e at 542°C, depicting increased interference to recrystallization due to polygonization
urring during dynamic annealing. (X750)

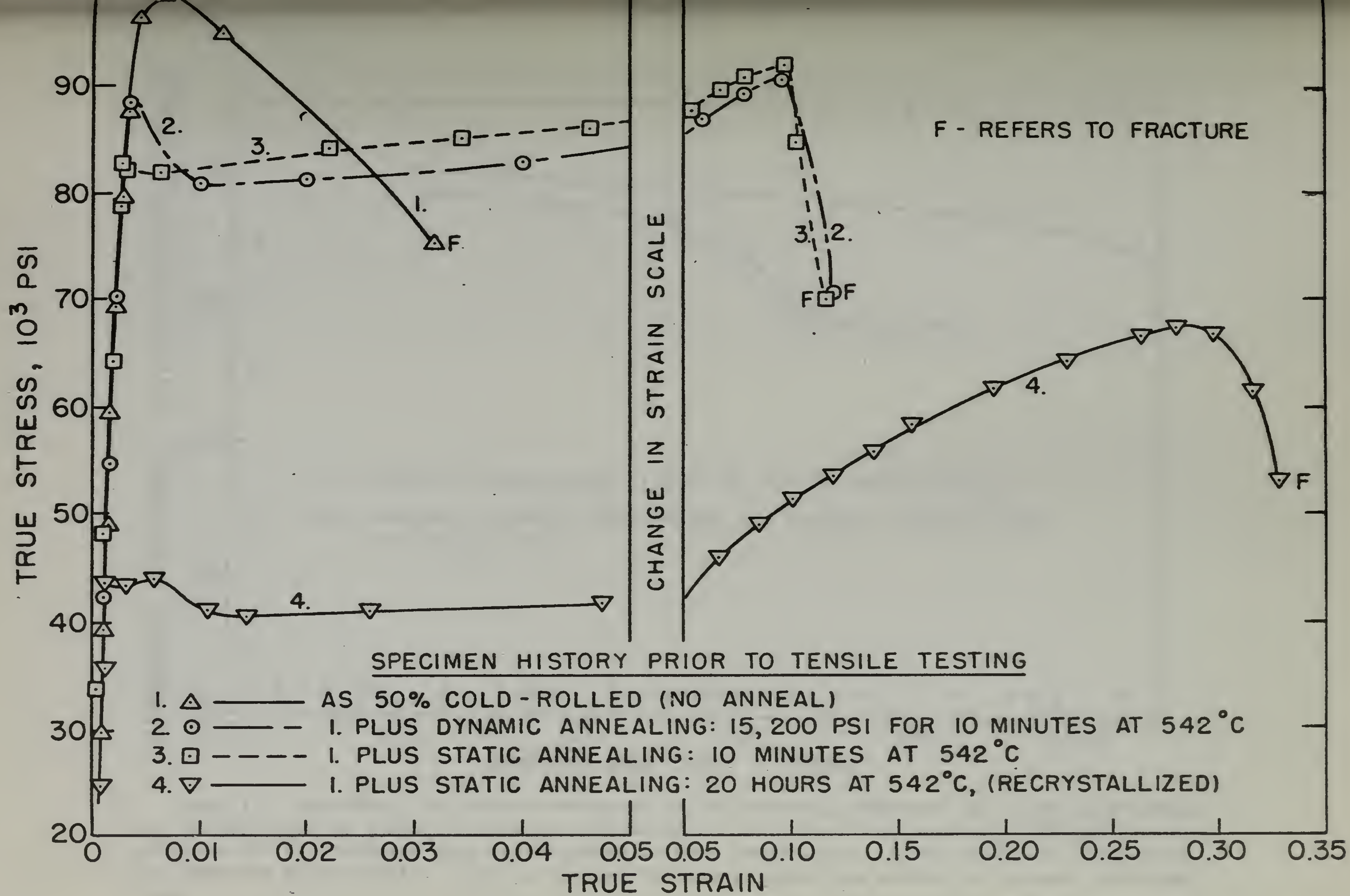


Fig.11 The effect of annealing conditions on the room temperature tensile characteristics of a previously 50% cold-rolled low carbon steel. Constant strain rate of approximately 2% per minute.

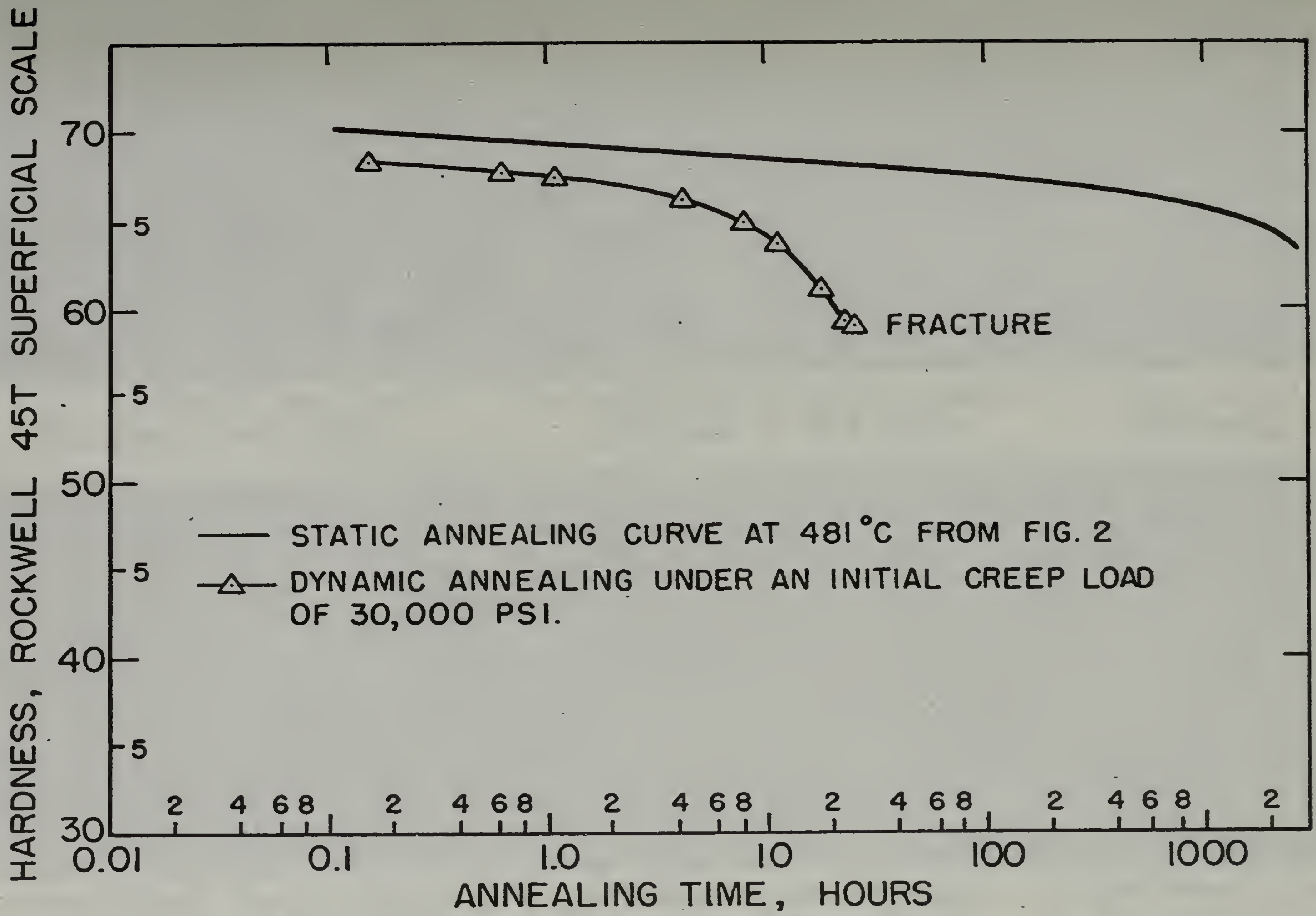


Fig. 12 The effect of dynamic annealing on the softening behavior for a 50% cold-rolled low carbon steel as compared to static annealing in the range where no recrystallization takes place at 481^{o}C. Hardness data are obtained at room temperature. Creep tests are interrupted for hardness measurements. Load is applied throughout annealing period for dynamic annealing test.

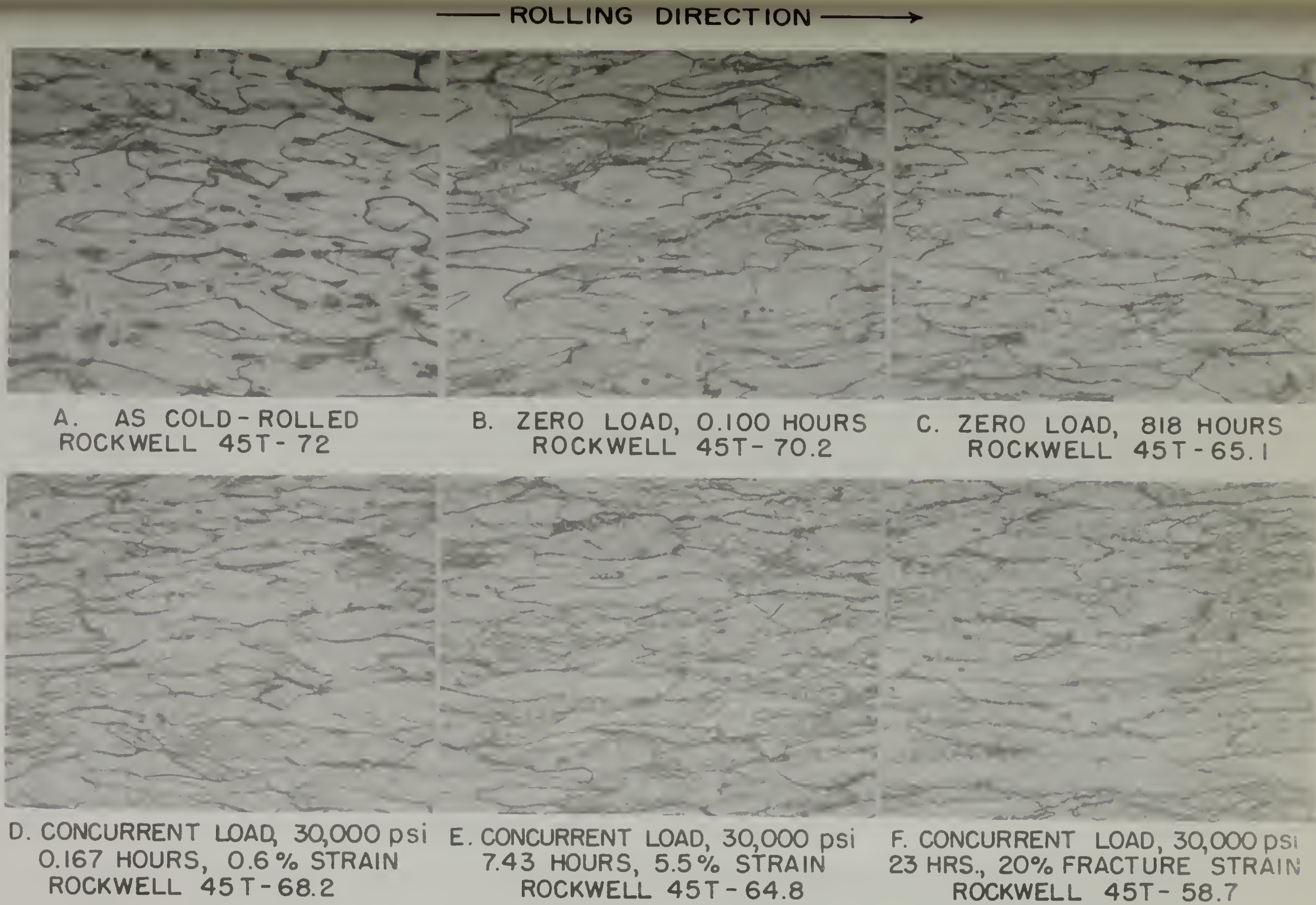


Fig.13 Photomicrographs showing the relative changes in microstructure as a result of static and dynamic annealing of a 50% cold-rolled low carbon steel in a range where recovery takes place at 481 ^{o}C during static annealing. (Hours refer to total time at temperature, X750)

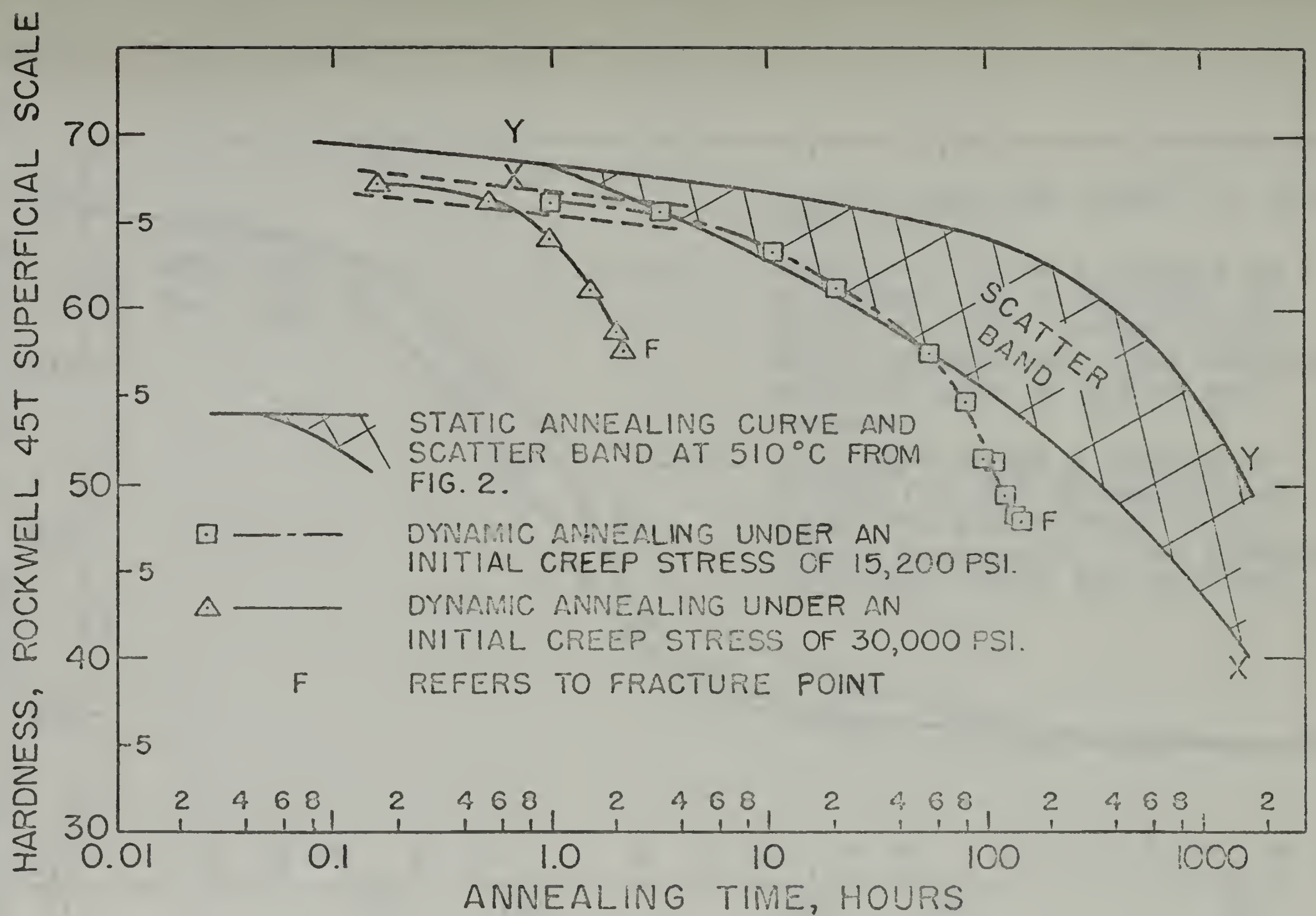


Fig. 14 The effect of dynamic annealing on the softening behavior for a 50% cold-rolled low carbon steel as compared to static annealing in the range where polygonization predominates at 510°C. Hardness data are obtained at room temperature. Creep tests are interrupted for hardness measurements. Load is applied throughout annealing period for dynamic annealing tests.

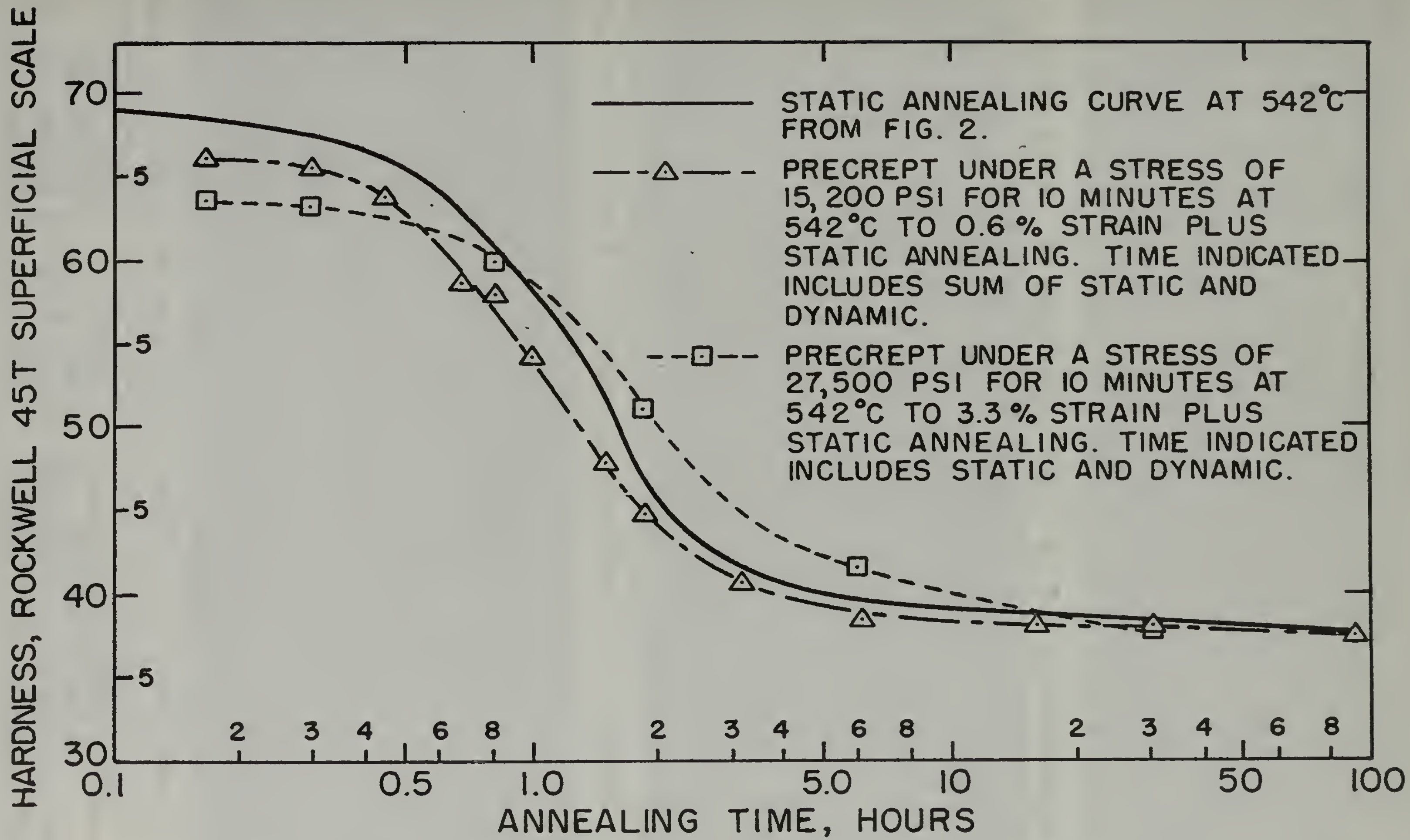


Fig. 15 The effect of prior dynamic annealing treatments on the softening behavior during subsequent static annealing for a 50% cold-rolled low carbon steel in the range where recrystallization predominates at 542°C under complete static annealing conditions. Hardness data are obtained at room temperature.

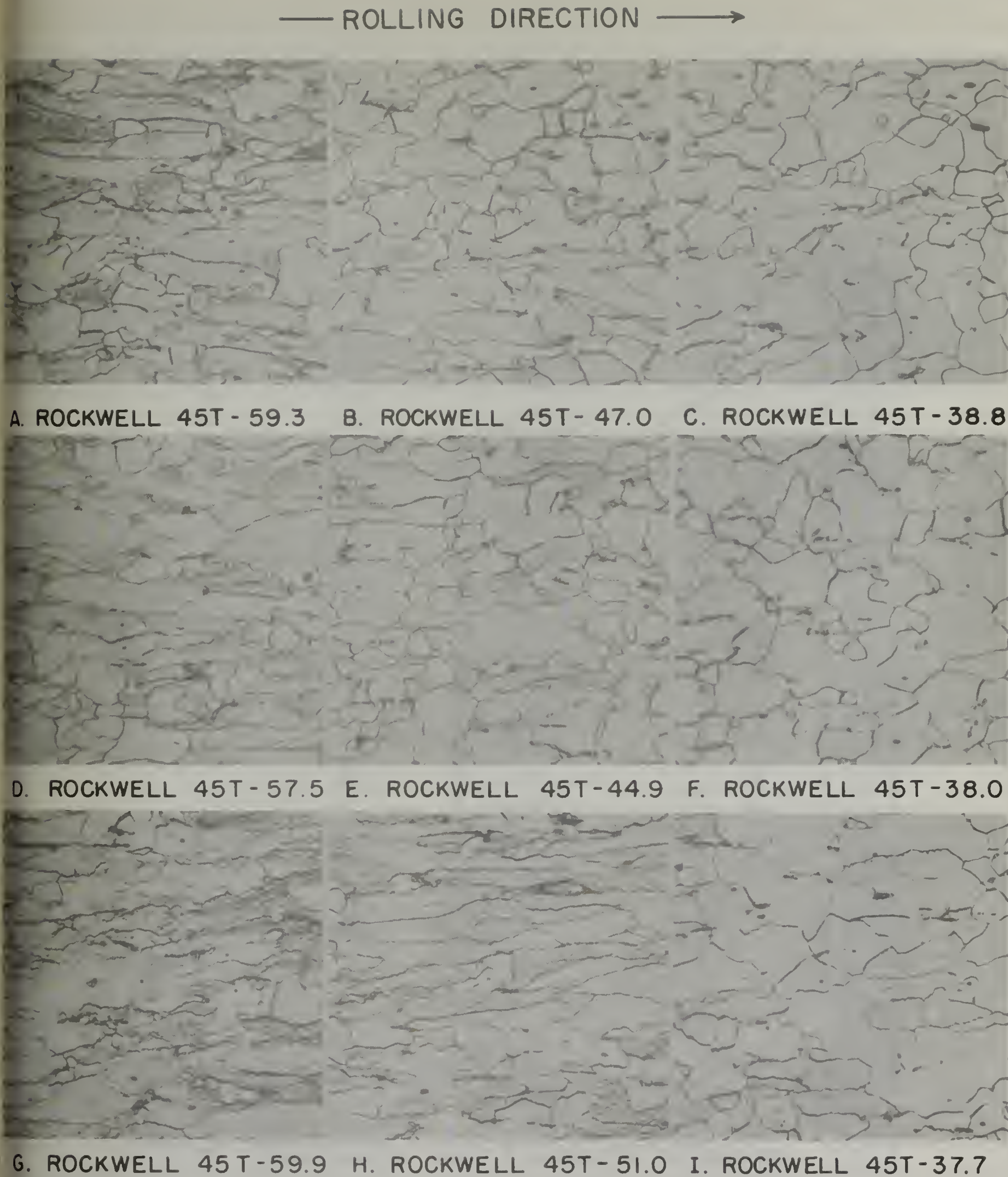


FIGURE	TOTAL TIME
A, D AND G	0.8 HOURS
B, E AND H	1.8 HOURS
C, F AND I	30.8 HOURS

FIGURE	CONDITION
A, B AND C	ALL STATIC ANNEAL
D, E AND F	PRECREPT AT 15,200 psi FOR 10 MIN.
G, H AND I	PRECREPT AT 27,500 psi FOR 10 MIN.

Fig.16 The Effect of prior dynamic annealing, of a 50% cold-rolled low carbon steel, on the microstructures obtained as a result of subsequent static annealing as compared to completely static-annealed specimens at a temperature where recrystallization predominates. All treatments at 542°C. (X750)

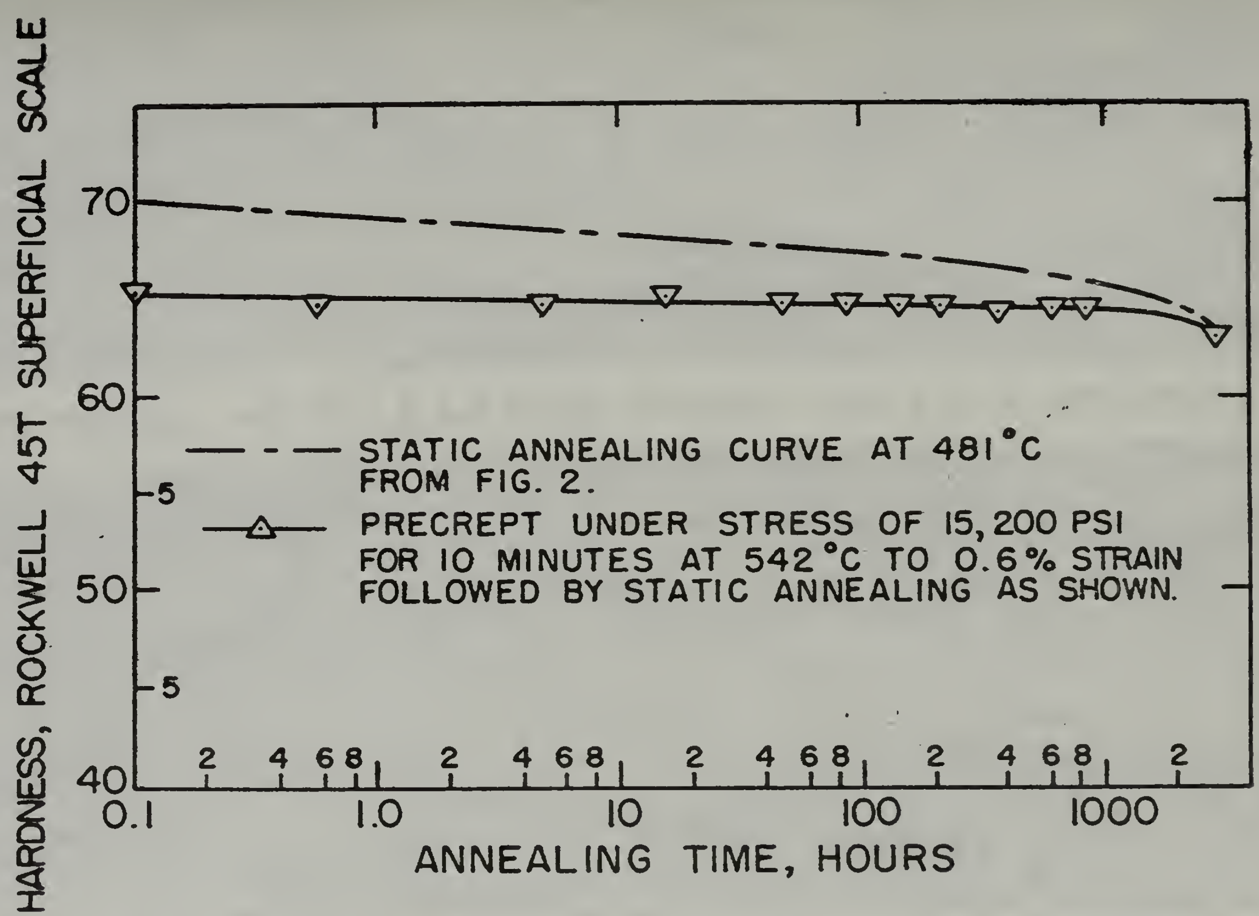
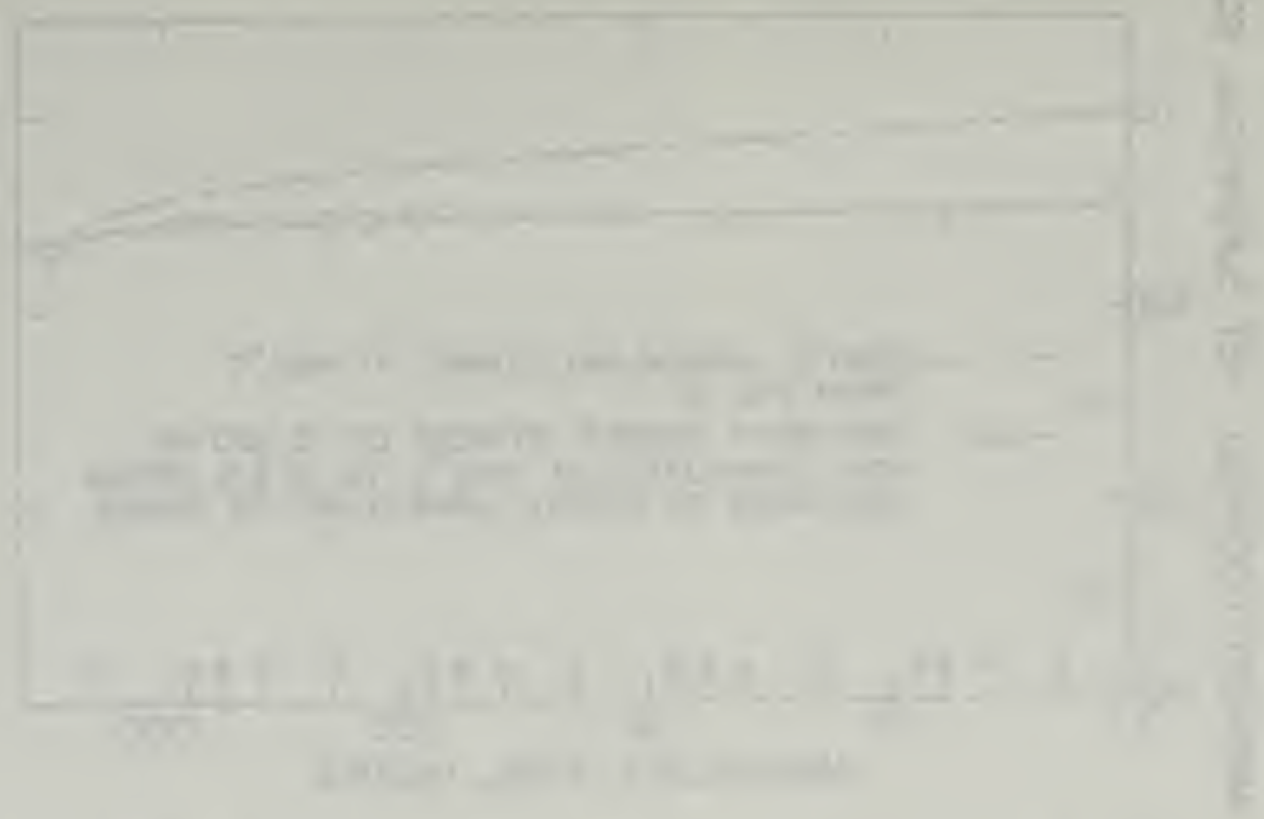
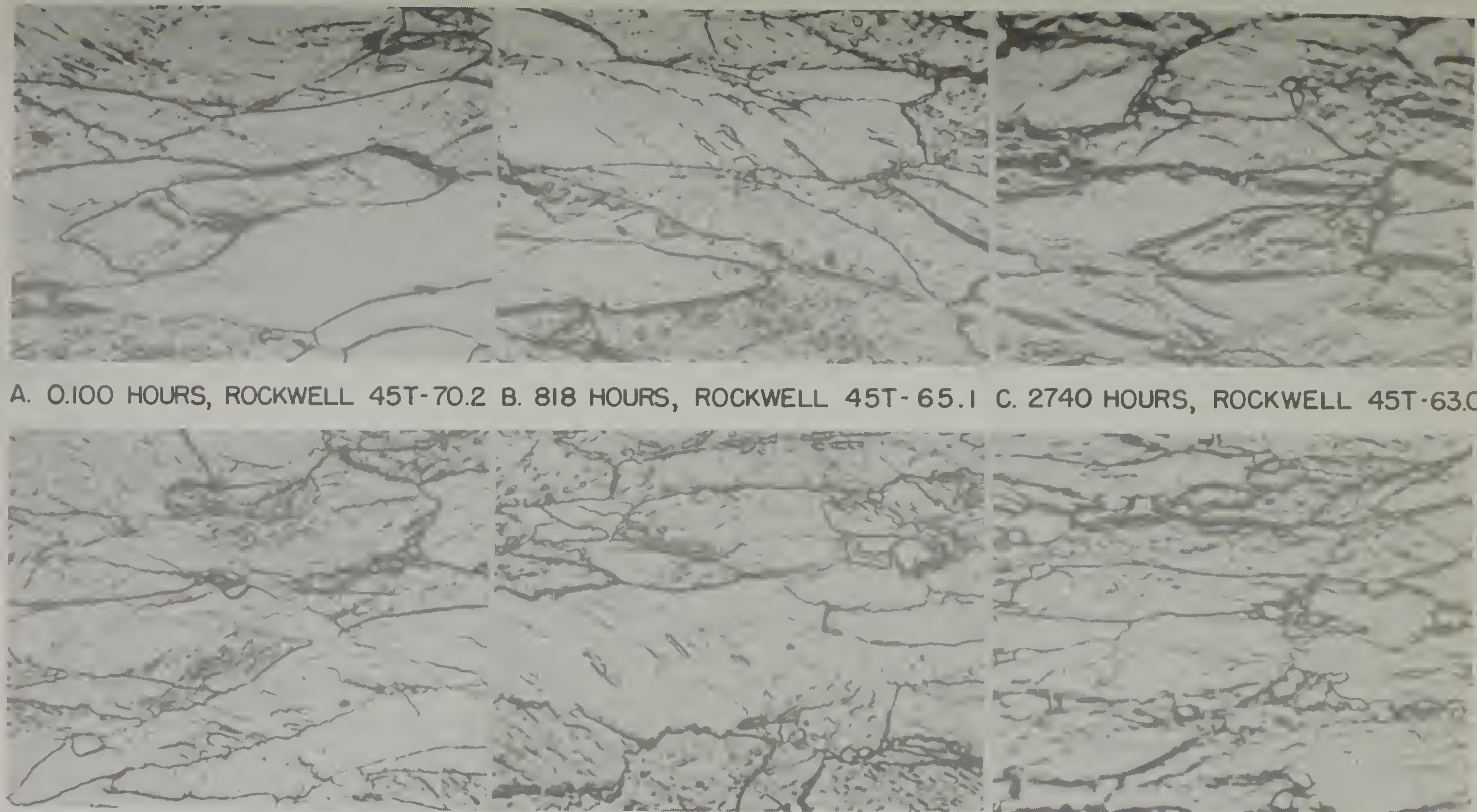


Fig.17 The effect of a prior dynamic annealing treatment at 542°C on the softening behavior during subsequent static annealing for a 50% cold-rolled low carbon steel in the range where recovery predominates at 481°C under complete static annealing conditions. Hardness data are obtained at room temperature.



——— ROLLING DIRECTION ———→



A. 0.100 HOURS, ROCKWELL 45T-70.2 B. 818 HOURS, ROCKWELL 45T-65.1 C. 2740 HOURS, ROCKWELL 45T-63.0

D. PRECREPT AT 15,200 psi FOR 10 MINUTES AT 542 °C FOLLOWED BY 0.100 HOURS, ROCKWELL 45T-65.2
E. PRECREPT AT 15,200 psi FOR 10 MINUTES AT 542 °C FOLLOWED BY 818 HOURS, ROCKWELL 45T-64.3
F. PRECREPT AT 15,200 psi FOR 10 MINUTES AT 542 °C FOLLOWED BY 2740 HOURS, ROCKWELL 45T-62.9

Fig.18 The effect of prior dynamic annealing at 542°C, of a 50% cold-rolled low carbon steel, on the microstructures obtained on subsequent static annealing as compared to completely static annealed specimens at a temperature where recovery predominates, at 481°C. (Hours refer to time at 481°C, X2000)

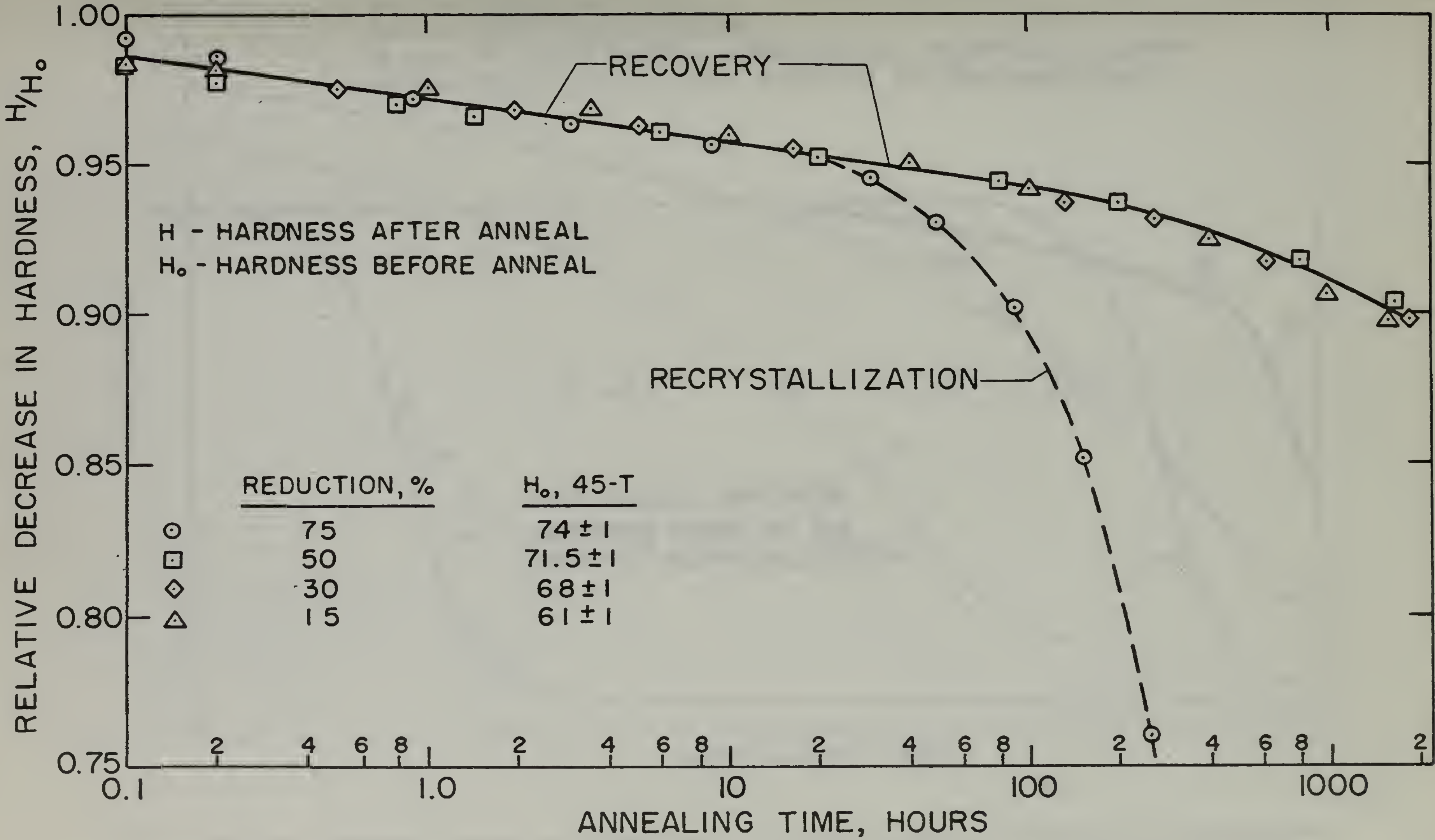


Fig. 19 The relative decrease in hardness as a function of time for four cold-rolled conditions of a low carbon steel annealed in a range where recovery predominates at 481°C.

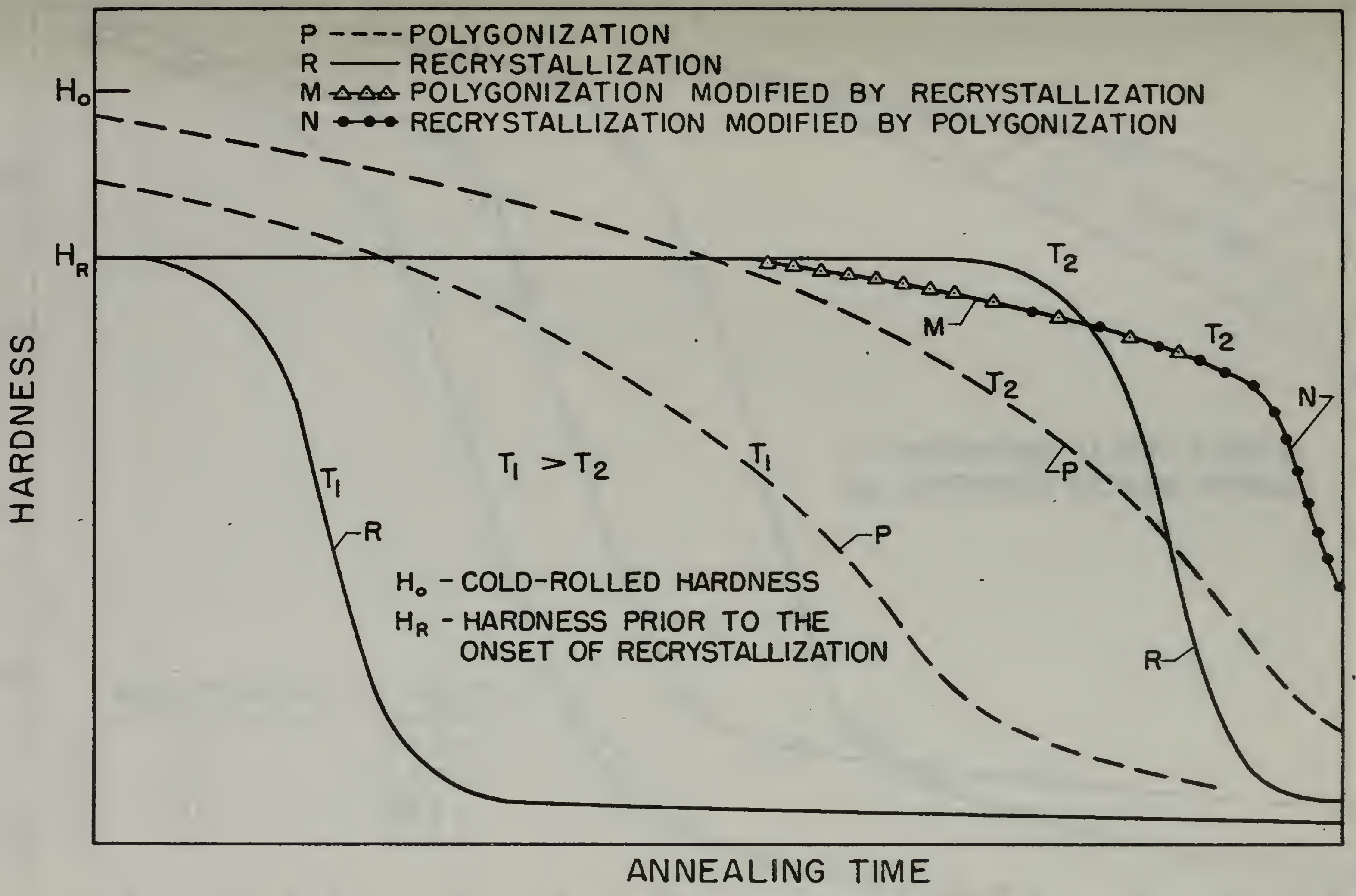


Fig. 20 Schematic annealing curves for a cold-worked metal illustrating the competitive roles played by recrystallization and polygonization at both a high temperature T_1, and at a relatively lower temperature, T_2.

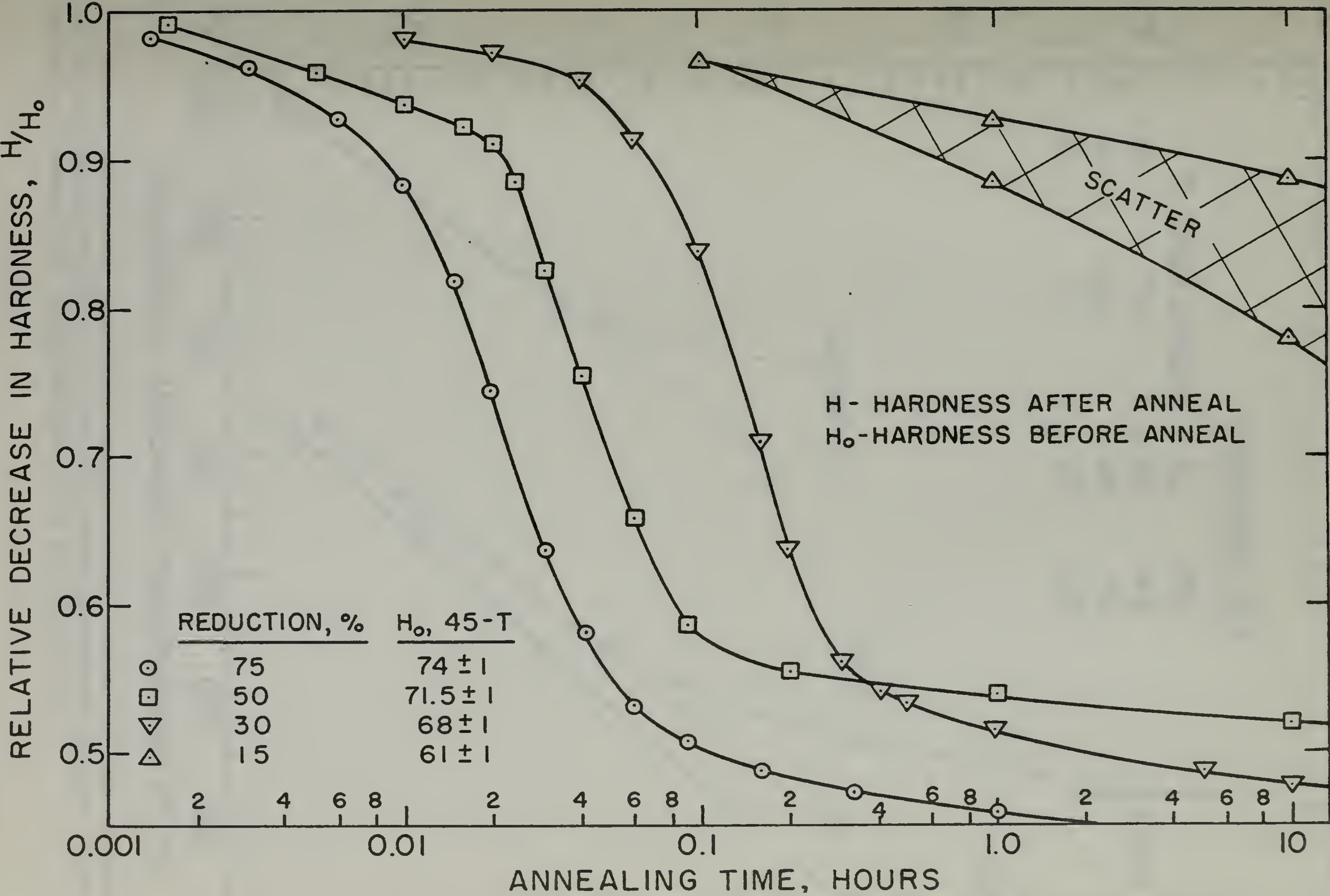
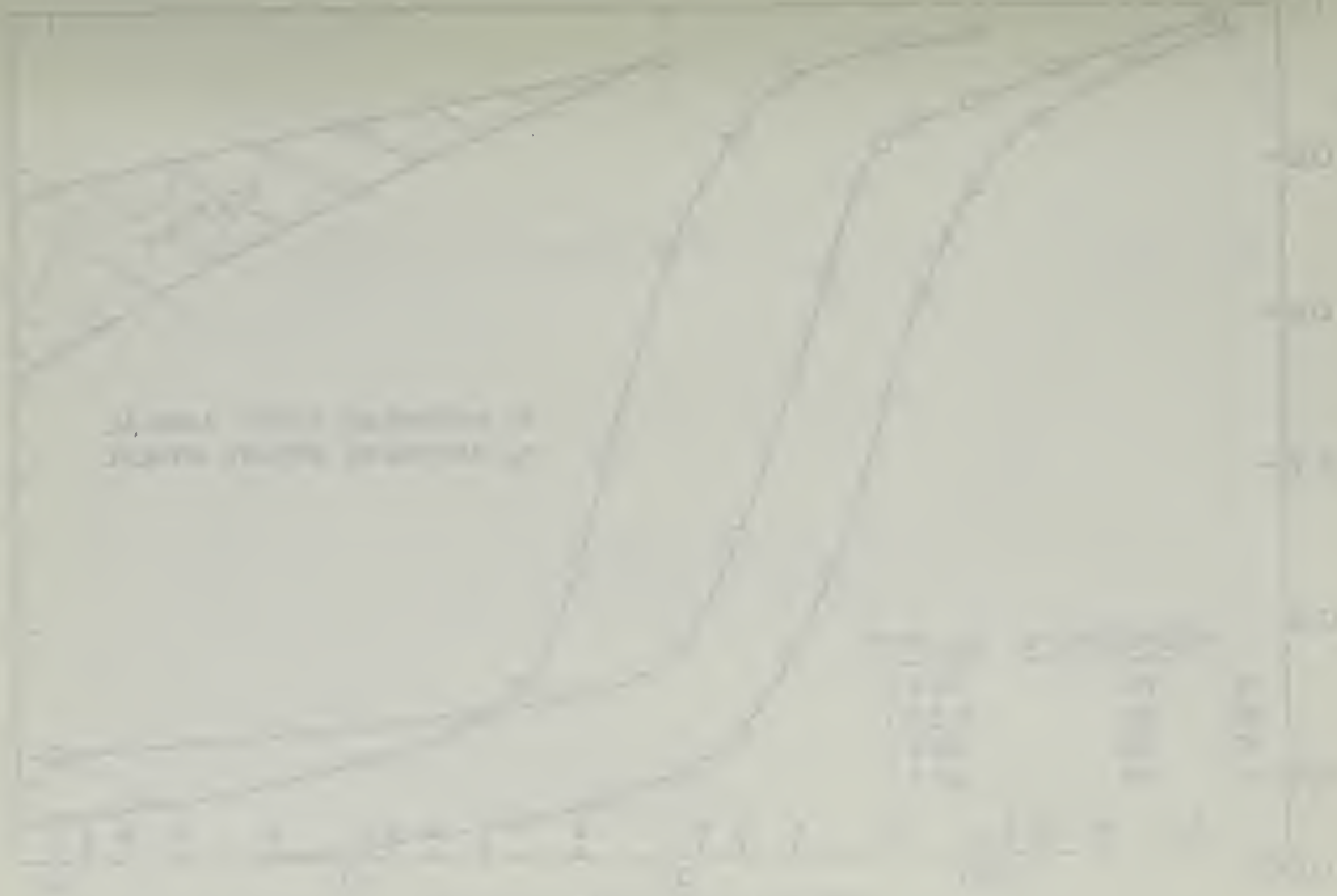


Fig. 21 The relative decrease in hardness as a function of time for four cold-rolled conditions of a low carbon steel annealed in a range where recrystallization predominates at 597°C.



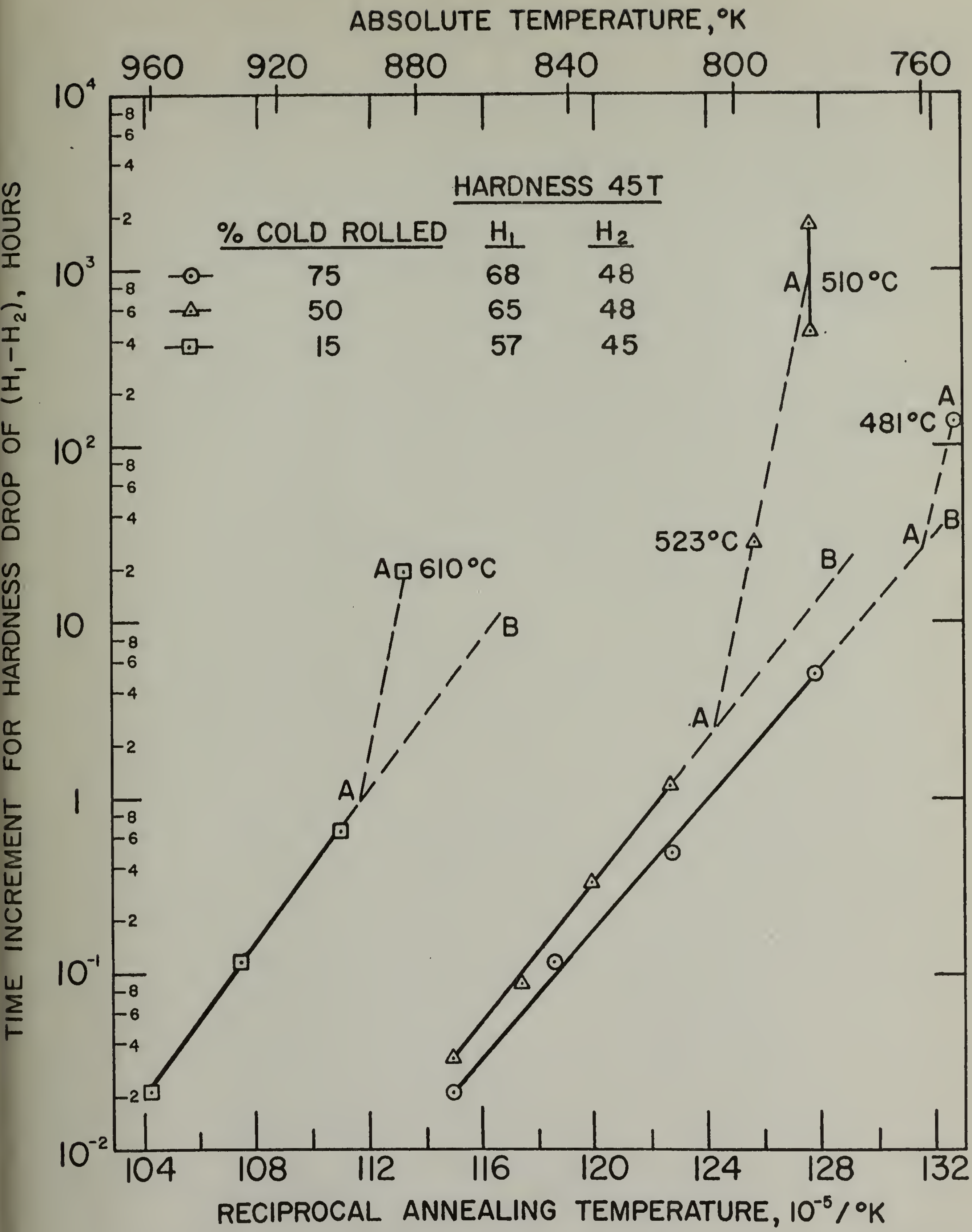


Fig.22 Reciprocal annealing temperature-time curves for the determination of the activation energies for three different cold-worked states. Time refers to interval for rapid hardness drop in Figs. 1-3.

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